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ALBERTA ENERGY AND NATURAL RESOURCES

# POPLAR CHEMICAL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

ALBERTA ENERGY AND NATURAL RESOURCES

# POPLAR CHEMICAL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY

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# EXECUTIVE SUMMARY

## INTRODUCTION

The Province of Alberta has a sizeable poplar resource, a part of which is suitable for the manufacture of conventional forest products. However, a significant portion is classified as unmerchantable for these uses but would be an ideal feedstock for a wood-based chemical industry. In addition, reject material and residues from merchantable sources can yield commercial volumes of chemicals at some of the larger existing forest product processing plants. Studies have shown that at least three sites in Alberta have access to a poplar resource adequate for supporting large wood-chemical facilities.

With the objective of exploiting the Province's unmerchantable poplar resource, Alberta Energy and Natural Resources is contemplating building a facility to test, develop and demonstrate technologies for the conversion of wood to chemicals. The selected conversion route consists of:

- pretreatment of wood to achieve partial delignification;
- hydrolysis of wood cellulose and hemicellulose to sugars;
- conversion of sugars to ethanol, food yeast and other chemicals;
- recovery of high quality native lignin.

The plant is to be designed for ease of modification to permit testing of a large number of processes and to produce a variety of products. Once in place and operating, it is expected to stimulate research and industrial development in biotechnology and lignin processing in the province.

Alberta Energy and Natural Resources, through Alberta Research Council, have engaged W.L. Wardrop & Associates Ltd. to develop the conceptual design costs of such a facility. Assistance was provided by Morris Wayman Limited in technology assessment and selection and by Deloitte Haskins Sells Associates in defining management options.

## POTENTIAL BENEFITS OF THE FACILITY

The primary purpose of the facility is to develop technically, economically viable options for the utilization of Alberta's poplar resource. It will achieve this objective by demonstrating technologies for converting wood into its constituent components which, in turn, can be converted to chemicals. A number of such technologies and processes have been tested on bench and pilot scales. The next stage to achieve commercialization is demonstration on a relatively large scale under continuous operation such that technical problems can be resolved and economics established. The proposed facility will fulfill this crucial need.

It is expected that by demonstrating technologies to utilize unmerchantable poplar, the proposed facility will extend the development potential of the forestry sector to provide thousands more jobs in the future. Biological processes for enzyme production and fermentation will be incorporated into the facility to help stimulate growth in Alberta of industries based on biotechnology.

The proposed conversion route breaks down the wood into the following components:

1. Hexose sugars (cellulose)
2. Pentose sugars (hemicellulose)
3. Lignin

It is proposed that initially the hexose sugars be converted to ethanol and pentose sugars to food yeast. There are, however, a great many uses for these sugars, especially as a source of fermentation products, including: butanol; acetone; penicillin and other antibiotics; enzymes; and vitamins. It has been claimed that a major fraction of U.S. chemical industry can be based on four fermentation products: ethanol; isopropanol; n-butanol; and 2, 3 butanediol. Thus, although ethanol may be the primary product of this plant, it is not the only way to make use of Alberta's poplar resources.

Lignin, a naturally occurring phenolic polymer, is presently being recovered from sulphite liquor and used for the production of vanillin, drilling mud additives, water treatment chemicals; as a dispersant for concrete and dye stuff; and as a binder for metal ores and animal feed. High quality native lignin, recovered during the conversion of wood to chemicals, has a high potential for use as a raw material for the production of adhesives, resins, pharmaceuticals, and chemicals. Its potential conversion to phenol might be capable of opening up large markets for this wood component. Simultaneous research and development into lignin utilization would help develop large markets for this material. It is not inconceivable that lignin which is a byproduct of the cellulose to ethanol process, could in itself support a substantial chemical industry in Alberta.

| PRETREATMENT | ACID HYDROLYSIS | ENZYME PRODUCTION AND HYDROLYSIS | FERMENTATION | DISTILLATION | BY-PRODUCTS |
|---|---|---|---|---|---|
| AUTOHYDROLYSIS<br>Iotech (Masonite)<br>Stake<br>Wenger<br><br>ORGANOSOLV<br>(PASZNER)<br>(BENCH-SCALE ONLY) | MULTI-STAGE DILUTE ACID | BATCH ENZYME PRODUCTION IN STIRRED TANK REACTORS<br><br>HYDROLYSIS BY BATCH-FEEDING OR CONTINUOUS FEEDING OF SUBSTRATE TO ENZYME BROTH<br><br>SIMULTANEOUS SACCHARIFICATION FERMENTATION (ALTERNATE) | CONTINUOUS FERMENTATION IN STIRRED-TANK REACTORS WITH YEAST CYCLE<br><br>CONTINUOUS FERMENTATION IN TOWER FERMENTER | PROPRIETARY AZEOTROPIC DISTILLATION PROCESSES | LIGNIN RECOVERY BEFORE HYDROLYSIS<br><br>LIGNIN RECOVERY AFTER HYDROLYSIS<br><br>PENTOSE CONVERSION TO SINGLE CELL PROTEIN |

EXHIBIT 1: PREFERRED TECHNOLOGIES

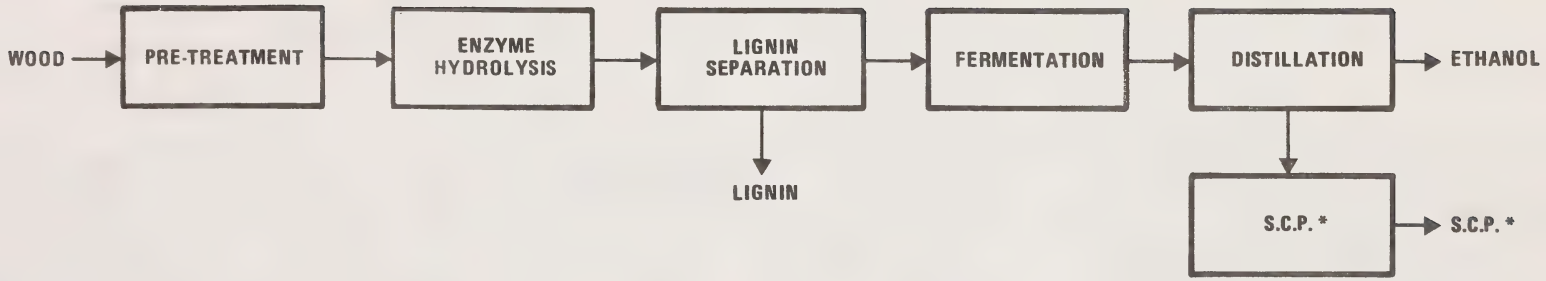
## SELECTED TECHNOLOGIES AND PROCESSES

Over the last few years, technologies for the conversion of wood to sugar have undergone rapid development. The Study Team initiated a survey of available literature and contacted organizations involved in research and process development in order to assemble comprehensive and up-to-date information on the work in progress. A number of technologies were selected for demonstration at the proposed facility and are summarized in Exhibit 1. Focus for technology development is considered to be in the areas of pretreatment, enzyme production and by-product recovery and processing. Conventional processes have been chosen for the remaining steps such as fermentation and distillation.

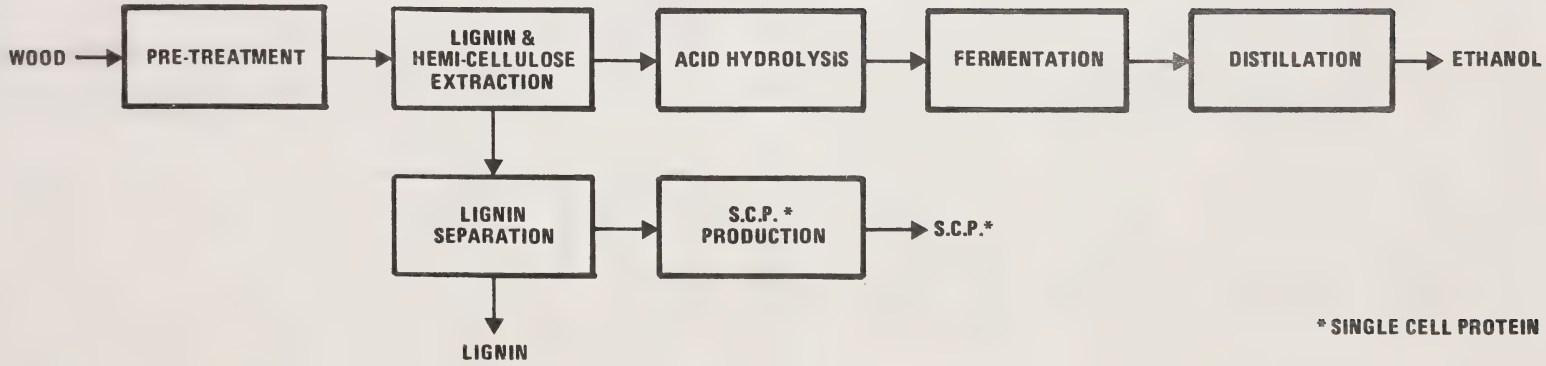
Primary process configurations, which are composed of the preferred technologies, are presented in Exhibit 2. The primary enzymatic process makes ethanol from wood through minimum process steps, uses wood substrates to produce enzyme, and recovers high quality by-products. As lignin can be degraded upon contact with acid, it is extracted for the primary acid process before the hydrolysis step to preserve its quality.

The conceptual design provides for testing and demonstrating a number of other technologies and process configurations without major modifications to the proposed facility. This is essential in view of the rapid developments, especially in the areas of enzyme production and hydrolysis. Similarly, the fermentation systems can be used to convert sugars to products other than ethanol or yeast. Technologies to process lignin to high value products can be easily added when such processes are ready for industrial scale demonstration.

## ENZYMATIC HYDROLYSIS




## ACID HYDROLYSIS




* SINGLE CELL PROTEIN

EXHIBIT 2: PRIMARY PROCESSES

RESEARCH AND DEVELOPMENT

The function of the proposed facility will be to demonstrate technologies which have progressed through pilot stage and are ready for commercialization. However, it is recognized that a significant amount of basic research and bench-scale work will be required to further improve these technologies and to solve problems discovered during demonstration. Areas in which work is expected are: pretreatment; enzyme production; hydrolysis; lignin processing; and pentose fermentation. It may also be desirable to help develop some of the processes which are now in the laboratory, but have a high application potential in the long-term. An example of such processes is organosolv saccharification.

It is our opinion that most of the laboratory and bench-scale development work should be contracted out to local universities and research centres. This will have the following positive effects:

- It will provide the program managers with the flexibility to design the R&D program for maximum effectiveness, and will contribute to the utilization of the best expertise, regardless of where such expertise may be available.

- It will help limit the function of the proposed facility to essentially demonstration, thereby improving its effectiveness and reducing costs and manpower requirements. This will also encourage the private industry to use the facility for testing their processes as their technology ownership interests will be well protected.

- Research in biotechnology at local institutions will help develop centres of excellence in this "science of the future" and could spur growth of the bioindustry in the Province. Some Alberta universities and research centres are already planning developments in this area and this program will help them meet their objectives and vice versa.

Control laboratories to monitor process performance, and a bench-scale development area will be provided at the facility to ensure that the expertise available at the pilot plant is fully utilized to help develop and demonstrate wood conversion technologies.

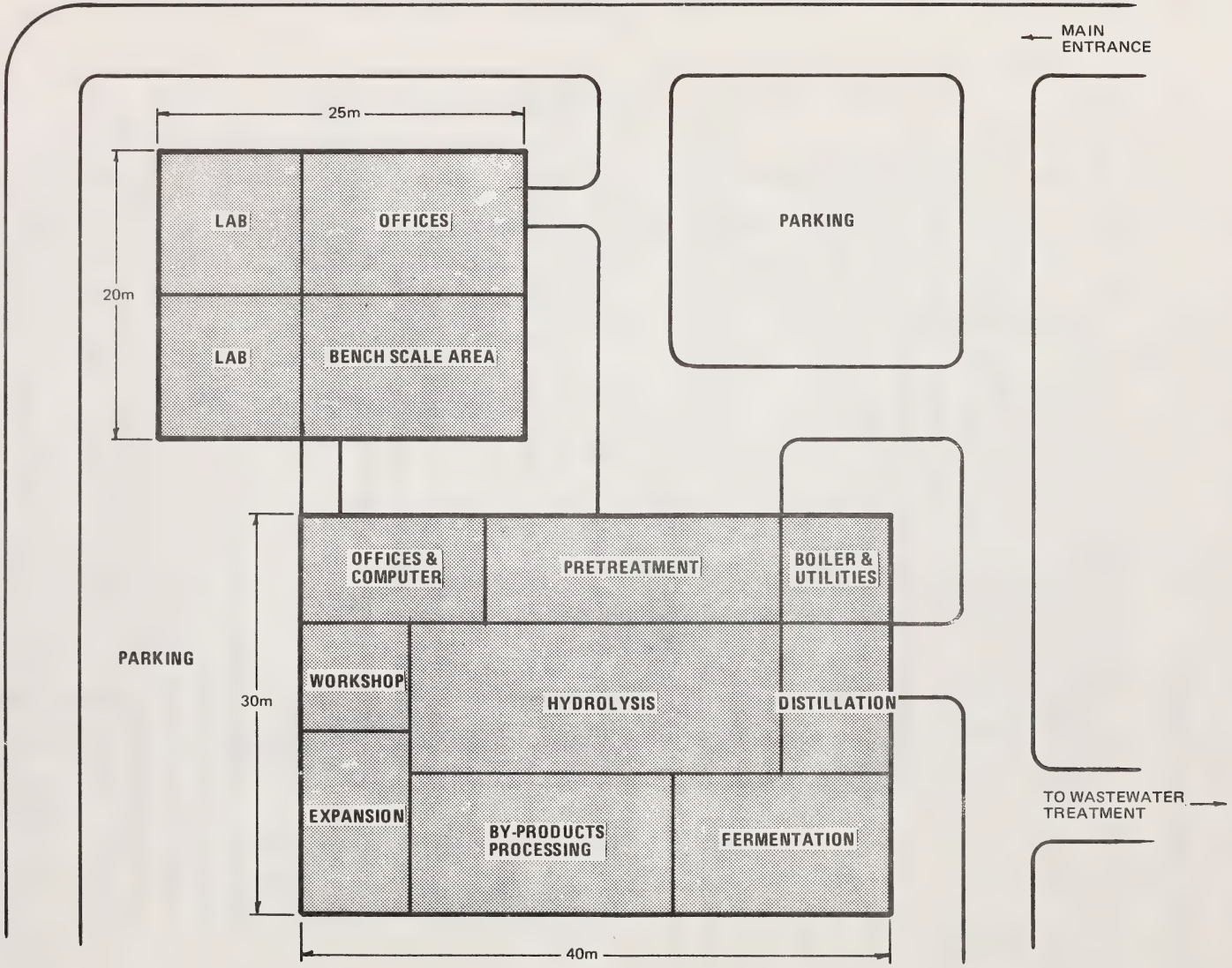



EXHIBIT 3: PLANT LAYOUT

PLANT CONCEPT

The proposed demonstration facility would process 10 tonnes/day of Bone Dry Wood Equivalent (BDWE). It is expected to operate 200 days per year at full utilization, allowing adequate time for process changeover and maintenance activities. Primary plant products are:

Ethanol = 475 tonnes/year
Lignin = 370 tonnes/year
Food yeast = 75 tonnes/year

A 10 tonnes/day BDWE capacity has been selected because it is considered to be the minimum desirable size for testing and demonstrating processes on a continuous basis, and obtaining scale-up data. A high level of process monitoring and data logging and processing capabilities are included in view of the nature of the facility.

Exhibit 3 presents the proposed plant layout. The main plant building occupies 1200 $m^2$ and includes all process units, utilities, computer, control room, workshop and offices for the operating staff. Space is available for additions to the various process systems, and for adding new systems. Administration and laboratories are located in an adjacent 500 $m^2$ building. The laboratories will have the following components:

- Chemical
- Microbiological
- Bench-scale Area

Analytical capabilities at the laboratory are designed to support plant operations and to assist in process development work carried out in the bench-scale area.

The plant would be located on a 10-acre site. Because of the small size of the facility, no major site selection criteria has been identified as crucial to the location of the facility. However, proximity to a major industrial centre will facilitate transfer of information and personnel exchange. Good "quality of life" is also important in site selection because highly trained personnel will operate the plant.

CAPITAL COSTS

The conceptual design capital cost for a 10 tonne/day BDWE capacity plant is estimated at $29.5 million in 1981 dollars. A detailed breakdown is presented in Exhibit 4. We expect the level of accuracy of our cost estimates to be consistent with the conceptual design stage at ±20 percent.

As detailed in the implementation schedule (Exhibit 5) the proposed facility would require 2 years to design, build and commission, and the bulk of the expenditures will occur during the second year. To ensure adequate provision from a budgeting standpoint, we recommend that an annual escalation of 14 percent be included in the cost. This will result in a budget figure of $37 million for the proposed facility.

As required by the Terms of Reference, the proposed facility has been designed to incorporate a high degree of flexibility for testing various processes and for producing a number of by-products. If reduction in capital costs is desirable, some of the process systems can be modified and the level of sophistication in process control and data logging reduced without seriously compromising the basic objectives of the facility. Estimated cost of a facility with a reduced scope and flexibility is $23.5 million in 1981 dollars. Revised budget allowance alternative for this case is $30 million.

EXHIBIT 4

DEMONSTRATION PLANT CAPITAL COSTS

| Direct Costs | 1981 $ |
|---|---|
| Site Work and Land | $ 300,000 |
| Serviced Building | 3,000,000 |
| Process Systems | 11,112,000 |
| Laboratory Equipment | 1,230,000 |
| Utilities (including off-site electrical) | 1,963,000 |
| Controls & Computer | 1,010,000 |
| Workshop and Office Equipment | 500,000 |
| Wastewater Treatment | 200,000 |
| Sub-Total | $19,315,000 |
| Contingency (20% TDC) | 4,785,000 |
| Total Direct Cost (TDC) | $24,100,000 |

| Indirect Costs | |
|---|---|
| Engineering | 2,400,000 |
| Construction Management | 1,200,000 |
| Commissioning, including Computer Software | 1,300,000 |
| Studies and Approvals | 500,000 |
| Total Indirect Costs | $ 5,400,000 |
| 1981 Construction Cost Estimate | $29,500,000 |

BUDGET ALLOWANCE (for design and construction during 1982-83) $37,000,000

OPERATING COSTS AND REVENUES

Estimated annual operating cost for a 10 t/d BWDE plant is $2.5 million in current dollars. This cost is based on 200 days/year production. The remaining days will be used for process changeovers, modifications and maintenance. It must be emphasized that these expenditures will be required only when the plant approaches full utilization. The costs can be broken down as follows:

| | |
|---|---|
| Wages | 53.0 percent |
| Raw Material | 3.0 percent |
| Chemicals | 9.0 percent |
| Utilities | 7.0 percent |
| Maintenance | 24.0 percent |
| Miscellaneous | 4.0 percent |

The plant will employ 40-43 persons at full utilization, and their wages form more than 50 percent of total operating costs.

Expected plant revenues from product marketing at full utilization could approach $400,000 annually, thereby reducing the annual operating cost to $2.1 million in 1981 funds.

The above operating costs do not include the management of the overall program and research and development funding. These costs will depend on the type of management structure and the scope of R&D program. For preliminary budgeting purposes, we would suggest $500,000 per year in 1981 funds. This will result in an annual cost in the range of $3.5 million in 1984 when the plant starts operation.

## MANAGEMENT OF THE R&D PROGRAM AND DEMONSTRATION FACILITY

The preferred approach to the management of the demonstration facility and the R&D program is a form of government-industry consortium. A detailed management structure can be developed once the nature and number of participants is known. The advantages of this approach are: high potential of commercialization of technologies developed and tested; high relevance of work performed at the pilot plant with industrial development in Alberta; and the reduction of the share of public costs for the facility construction and operation. Chemical, pulp and paper, wood products, and petroleum industries should benefit from participation in the proposed facility.

An alternative consists of management by a Working Group headed by AENR and composed of industry, government and research representatives. Alberta Research Council will manage the program on behalf of the Working Group, although the day to day operation of the demonstration facility could be contracted out to the industry.

A detailed management study is considered desirable to survey industry attitudes, to encourage participation and to develop a formal management structure. Major issues that must be considered during the study are:

- ownership and licensing of technology;
- potential conflicts between research functions and technology development functions of the proposed organization; and,
- cost sharing between government and industry.

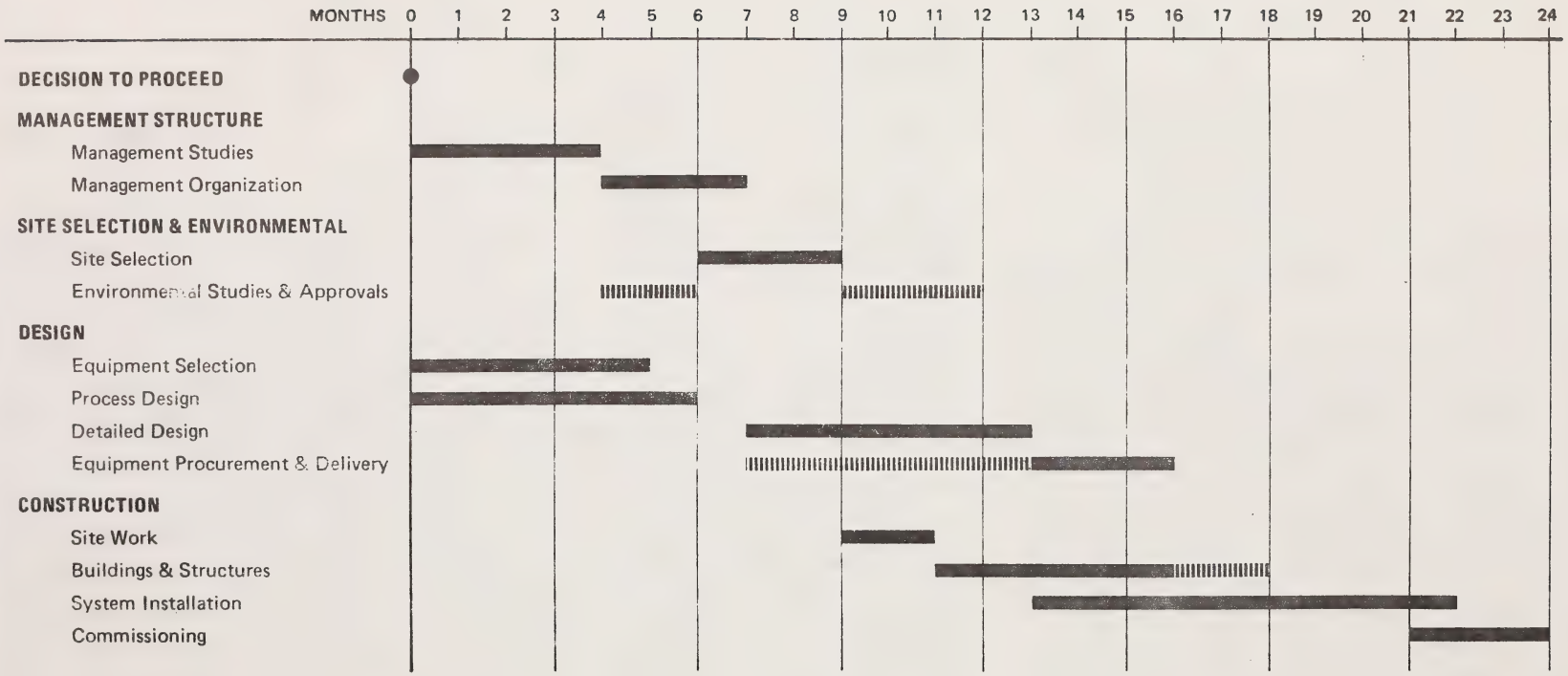



EXHIBIT 5: PROGRAM IMPLEMENTATION SCHEDULE

IMPLEMENTATION SCHEDULE

A 24-month implementation schedule is presented in Exhibit 5. Primary components are:

a) Management Structure Definition
b) Site Selection and Environmental Studies
c) Design
d) Construction

The proposed schedule assumes that the environmental assessment and site work can start immediately once a site has been selected.

We recommend the following first steps towards the development of the proposed facility:

1. Initiate a study to define the management structure.

2. Continue the equipment testing initiated during this study.

3. Commission the process design phase of the project.

Process design and equipment testing are the most crucial next steps in the development of design and costs of the facility.

PROVINCE OF MANITOBA
H. HUSAIN
March 19/93
REGISTERED ENGINEER

PROFESSIONAL ENGINEER ALBERTA
V. H. WIENS

W. L. WARDROP & ASSOCIATES LTD.

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THE ASSOCIATION OF PROFESSIONAL ENGINEERS, GEOLOGISTS and GEOPHYSICISTS OF ALBERTA
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W. L. WARDROP AND ASSOCIATES LIMITED

DETAILED DISCUSSIONS

BACKGROUND

## Introduction

The Department of Energy and Natural Resources of Alberta, acting through the Alberta Research Council, is planning to build a demonstration facility for making chemicals from poplar through pretreatment, hydrolysis and fermentation. Such an undertaking is part of a broader program the purpose of which is to open up the options for utilization of Alberta's extensive poplar resource. While the facility will produce ethanol, the focus will be on the technology of conversion of wood to industrial glucose and other non-saccharides.

The facility would be sized such that the experience of its operation can provide a basis for design of a full scale industrial plant. Since there is no suitable North American industrial experience, or even large scale pilot plant experience for the production of alcohol from wood, it will be equipped to demonstrate, on a continuous basis, the most promising processes, including both enzymatic and acid hydrolysis. The plant will be well instrumented and data logging and analysis capabilities provided to generate scale-up data. The accompanying laboratory will be capable of testing out on a smaller scale, other new processes not provided for in the plant itself.

In order to initiate this program, W.L. Wardrop & Associates Ltd. have been engaged to prepare a conceptual design report, including a review of the state of the art, the selection of the plant systems to be installed, estimation of the costs of construction and operation, some concepts of how the facility may be managed, review of markets for the products, and the development of a research and development program. The Terms of Reference for the study are presented in Appendix A. This report embodies the work carried out by Wardrop, and their subconsultants, Morris Wayman Limited and Deloitte Haskins & Sells Associates.

## Benefits of the Proposed Facility

The primary purpose of the facility is to produce technically, economically viable options for the utilization of Alberta's poplar resource. It will achieve this objective by demonstrating technologies for converting wood into its constituent components which, in turn, can be coverted to chemicals. The plant will incorporate biological processes for enzyme production and fermentation, thereby stimulating the growth in Alberta of industries based on biotechnology.

The end result will be the development of technologies to utilize unmerchantable poplar thereby extending the development potential of the forestry sector to provide thousands of more jobs in the future.

The proposed conversion route breaks down the wood into the following components:

1. Hexose sugars (cellulose)
2. Pentose sugars (hemicellulose)
3. Lignin

It is proposed that initially the hexose sugars be converted to ethanol and pentose sugars to food yeast. There are, however, a great many uses for these sugars, especially as a source of fermentation products:

i) fermentation industries for chemical manufacture. It is reported that 27 chemicals are now being made from biomass by fermentation.

ii) production of food and feed yeast.

iii) enzyme production essential for food processing and for the conversion of cellulose and starch to useful products.

iv) fermentation production of antibiotics, an essential component of health care, whose manufacture has been abandoned elsewhere in Canada.

A thorough review of "Biomass as a Source of Chemical Feedstocks" was recently (July, 1981) reported by B.O. Palsson, S. Fathi-afshar, D.F. Rudd and E.N. Lightfoot, University of Wisconsin, in Science. It was concluded that the raw materials and technology exist for basing a major fraction of the U.S. chemical industry on four fermentation products: ethanol, isopropanol, n-butanol and 2, 3-butanediol. The primary route for the introduction of these raw materials would be dehydration to olefins, which would provide a smooth transition to renewable feedstocks. However, to make these materials attractive as feedstocks or intermediates in chemical production, their current prices would have to be substantially reduced, to about 20-40% of present prices. These prices are of course relative to the petrochemical feedstock prices, and fermentation chemicals should become attractive as petroleum costs increase.

Brazil is a country where some of these products are already being manufactured from fermentation ethanol. In 1975, 416 million litres of ethanol were produced, mostly for use as automotive fuel. However, 14 percent or 57 million litres, were used by the chemical industry. By 1985 ethanol production will amount to 11.4 billion litres of which 10 percent, or 1.14 billion litres, will be used by the chemical industry. The Brazilian chemical utilization is largely through dehydration of ethanol to either ethylene or acetaldehyde, and then rebuilding other chemicals such as polyethylene, styrene, vinyl derivatives, acetic acid, higher alcohols such as butanol and ethylhexanol, butadiene, diethyl ether, ethylene glycol and its ethers, and ethyl halides.

Lignin, a naturally occurring phenolic polymer, is presently being recovered from sulphite liquor and used for the production of vanillin, drilling mud additives, water treatment chemicals and as a dispersing agent for concrete. High quality native lignin, recovered during the conversion of cellulose to ethanol, has a high potential for use as a raw material for the production of adhesives, resins, pharmaceuticals, and chemicals. Its potential conversion to phenol might be capable of opening up large markets for this wood component. Simultaneous research and development into lignin utilization would help to develop a lignin based industry in Alberta. It is not inconceivable that lignin which is a byproduct of the cellulose to the ethanol process could in itself support a substantial chemical industry in Alberta.

## Alberta's Poplar Resources

The requirement for this demonstration ethanol facility is based upon the availability of a very large unutilized poplar resource in Alberta. The concept of "availability" encompasses several components, especially quantity, quality and cost. These elements are well known to the Alberta Energy and Natural Resources (AENR), and will only be briefly summarized here.

The provincial net allowable annual cut of poplar, as measured from the viewpoint of the existing forest industries, is approximately 5 million tonnes (t) bone dry wood equivalent (BDWE). This figure refers to normal scaling practice. Considered from the viewpoint of a chemical plant, where whole trees are used, and where much of the cull wood is quite satisfactory as feedstock, the available annual cut could be in the order of 7 to 8 million tonnes. Not all of this may be readily accessible at acceptable harvesting costs, a factor which may lead to the reduction of the total available to the original figure of 5 million tonnes.

Specific locations identified as having sufficient poplar resource to support a large scale chemical plant are High Prairie, Slave Lake and Whitecourt, each with annual cut of over 500,000 tonnes of BDWE. However, these can be augmented by sawmill residues. For example, in the area surrounding High Prairie, sawmill residues amount to almost 1 million dry tonnes annually. At Grande Prairie, a combination of mill residues and standing poplar can provide about 500,000 tonnes of BDWE annually. Thus in the areas named, where poplar is relatively dense and accessible, there are about 3 million tonnes of poplar available annually. The recent report "Energy and Chemicals from Wood" by AENR (1) states that "three other sites, High Level, Manning and Lac La Biche, have sufficient gross volume to support large plants for up to 40 years, but are unlikely to be able to do so on a sustained yield basis without increasing hauling distances significantly, or harvesting immature trees."

If we include in our poplar resource the readily accessible allowable annual cut corrected for usable cull and whole tree harvesting, plus sawmill residues, a figure of about 5 million tonnes per year can be considered realistic. At 300 litres of ethanol per tonne, this is adequate to produce 1,500 million litres per annum (L/a) of ethanol. A single, full-size industrial plant could produce 50 million L/a. Therefore, the present accessible poplar resource is adequate feedstock to supply 30 full size ethanol plants.

The report mentioned above quoted the delivered cost of poplar roundwood at $35 per tonne of BDWE and mill residues at $2-$17/t depending upon hauling distance, averaging about $13/t. A mix of half standing poplar and half sawmill residues should then have a delivered cost of $24/t, or 8¢/L of ethanol. This is an acceptable cost.

| PRETREATMENT | ENZYME PRODUCTION | ENZYMATIC HYDROLYSIS | BACTERIAL CONVERSION | ACID HYDROLYSIS | FERMENTATION | DISTILLATION ETHANOL SEPARATION | BY-PRODUCTS |
|---|---|---|---|---|---|---|---|
| Auto-hydrolysis | Natick (T.reesei) | Natick | MIT | Single-stage high temp., dilute acid | Saccharomyces cerevisiae | Azeotropic | Lignin separation before hydrolysis |
| Mechanical | Gulf (T.reesei, no purification) | Gulf (SSF) | GE | Strong Acid (with or without acid recycle) | S.pombe | Extractive | |
| Organolsolv Pulping & Saccharification | NRC (Thermophilic bacteria) | Plug flow reactor | Clostridium thermocellum | | S.guilliermondi | Molecular Sieve | Lignin removal after hydrolysis |
| Solubilization & Swelling of Cellulose | | Lawrence Berkeley (Enzyme Recycle) | Clostridium thermo saccharolyticum | Multi-stage Dilute Acid | Zymomonas mobilis | Membrane | |
| Acid Pretreatments<br>Strong Acid<br>Dilute Acid | | Immobilized Enzymes | | Dilute Acid with Solids recycle | Batch<br>Continuous Tower<br>Vacuum<br>Biostill | | Pentose conversion to<br>• Butanol<br>• SCP<br>• Molasses |
| Other<br>• Radiation<br>• Thermal<br>• Microbial Lignin Degradation<br>• Pyrolysis<br>• Wet oxidation<br>• Ultrasonic | | | | | Pachysolen tannophilus (Pentose Fermentation)<br>Isomerization-Saccharomyces cerevisiae (Pentose Fermentation) | | |

**OVERVIEW OF CELLULOSE CONVERSION TECHNOLOGY**

**TABLE 1**

TECHNOLOGY REVIEW

## Introduction

An extensive literature survey has been performed to determine the extent of work being done in ethanol production from cellulose. This survey has been supplemented by discussions with researchers, process engineers and plant operators, and by visits to locations where work is being performed in crucial areas of cellulose conversion.

Results of our findings, with assessments and preliminary selection of technologies are presented in Appendix B under the following headings:

- Pretreatment
- Acid Hydrolysis
- Enzymatic Hydrolysis
- Bacterial Conversion of Cellulose to Ethanol
- Fermentation
- Distillation - Ethanol Separation
- Pentose Fermentation
- By-Products

Reports on site visits and meetings with researchers are presented in Appendix C.

## Summary of Technology Review

Table 1 presents an overview of the various processes and technologies. A general conclusion is that processes which are highly attractive from the standpoints of yields and economics are now on the horizon and that a demonstration facility will greatly hasten their commercialization. Specific conclusions follow:

1. Perhaps the single most important development in cellulose conversion technology is the advent of autohydrolysis. This has brought back into the picture processes which in the past were not technically or economically feasible. Autohydrolysis systems are being offered by Stake Technology Ltd., Iotech Corporation and Wenger Manufacturing, Inc. In these processes, wood is treated to a high pressure and

temperature and then rapidly exposed to the atmosphere. Results are partial hydrolysis of hemicelluloses, disintegration of the lignocellulosic bond, and conversion of chips into a powder. Iotech and Stake processes use high pressure steam. Wenger extruder requires only power input.

2. Other pretreatment methods are expected to be unable to compete with autohydrolysis from the standpoints of product quality, costs and energy consumption.

3. A method which extracts lignin and saccharifies wood simultaneously is the organosolv pulping and saccharification process originally proposed by Kleinart. Problems have included low sugar yields and difficulties in obtaining pure lignin. Dr. Laszlo Paszner at the University of British Columbia claims that these problems have been overcome by the use of a special solvent. However, the process requires further development on a bench-scale before it is ready for demonstration.

4. Drastic improvements in enzyme production have been achieved over the past few years. These have been achieved through mutation of Trichoderma viride to isolate active strains. However, problems in achieving balanced enzyme activity remain and must be addressed to reduce costs. Enzyme production should be a key area addressed by the proposed facility.

5. Direct bacterial conversion of cellulose to ethanol is being developed at Massachusets Institute of Technology (MIT) but significant increase in activities and conversion rates is required before a commerical process can be developed.

6. A number of acid hydrolysis schemes are under development and efforts now appear to focus on applying these techniques to wood substrates obtained by autohydrolysis. Conventional processes are not expected to be competitive in the market place because of low yields and high capital costs.

7. The most active area of research in fermentation is xylose conversion to ethanol using Pachysolen tannophilus. Substantial work is required before this can be integrated with hexose fermentation to result in an economic and energy efficient process. Xyloses can also be fermented using the brewer's yeast after enzymatic isomerization to xylulose. However, product inhibition restricts such isomerization, and further research is required.

8. Most of the hexose fermentation research is aimed at grain or sugar based processes, and once developed, these technologies can be adopted by wood alcohol plants. As the focus of development at the demonstration facility is to be pretreatment and hydrolysis, continuous fermentation using Saccharomyces cerevisiae is selected.

9. Ethanol separation by distillation-dehydration is a well proven technique. New energy efficient approaches are being developed and should find application first in the grain and sugar ethanol industries. It is our opinion that these new processes need not be tested at the proposed facility.

10. By-products, especially lignin are expected to be the key to the economics of ethanol production from wood. Techniques are now available which recover this material in a native form as part of the conversion process. Research and development on manufacturing secondary products from lignin continues. This is another high priority area in ethanol production from wood.

11. Pentoses present in the wood can be converted to chemicals other than ethanol such as butanol, and single cell protein. Technology for such conversions is well established. Single-cell protein production is preferred for the demonstration plant because of the very high market potential. However, the fermentation system provided for single-cell protein production should also be suitable for butanol fermentation.

| PRETREATMENT | ACID HYDROLYSIS | ENZYME PRODUCTION AND HYDROLYSIS | FERMENTATION | DISTILLATION | BY-PRODUCTS |
|---|---|---|---|---|---|
| AUTOHYDROLYSIS<br>Iotech (Masonite)<br>Stake<br>Wenger<br><br>ORGANOSOLV<br>(PASZNER)<br>(BENCH-SCALE ONLY) | MULTI-STAGE<br>DILUTE ACID | BATCH ENZYME PRODUCTION IN STIRRED TANK REACTORS<br><br>HYDROLYSIS BY BATCH-FEEDING OR CONTINUOUS FEEDING OF SUBSTRATE TO ENZYME BROTH<br><br>SIMULTANEOUS SACCHARIFICATION FERMENTATION (ALTERNATE) | CONTINUOUS FERMENTATION IN STIRRED-TANK REACTORS WITH YEAST CYCLE<br><br>CONTINUOUS FERMENTATION IN TOWER FERMENTER | PROPRIETARY AZEOTROPIC DISTILLATION PROCESSES | LIGNIN RECOVERY BEFORE HYDROLYSIS<br><br>LIGNIN RECOVERY AFTER HYDROLYSIS<br><br>PENTOSE CONVERSION TO SINGLE CELL PROTEIN |

TABLE 2

SYSTEM SELECTION

This activity can be broken down into three parts:

1. Selection of appropriate technologies.
2. Formulation of alternative process configurations.
3. Selection of process configurations.

A key criterion used throughout the selection process is compatibility with the Terms of Reference and objectives of the facility. Specific criteria are defined for each technology and process configuration to assist in the selection process.

Technology Selection

Table 2 summarizes the technologies selected for the demonstration and bench-scale testing at the proposed facility. Selection criteria and evaluations for each technology follows:

Pretreatment

The major selection criteria for the pretreatment processes are:

- The pretreatment process should have proven effectiveness in terms of improving cellulose sensitivity to hydrolysis.

- It should allow recovery of by-products as high value commodities.

- It should have been demonstrated on a pilot or production scale.

Of the various processes, autohydrolysis systems, especially Stake and Iotech, meet all of the above characteristics. The effectiveness of the Wenger extruder was established by tests on Alberta poplar wood. The pulp was tested in Professor Wayman's laboratories in Toronto and results comparable to Stake and Iotech pulp were obtained. These results are presented in Appendix D. The process does not require high pressure steam and capital costs are low. The three foregoing processes are recommended for application at the demonstration plant.

Another process which meets the first two criteria is the organosolv saccharification process developed by Dr. Laszlo Paszner at the University of British Columbia. It performs three crucial functions, namely: pretreatment; lignin separation; and saccharification in a single step. However, the process has yet to be demonstrated on a pilot scale to confirm yields and establish costs and economics. This process is an excellent candidate for bench-scale testing at the proposed facility.

None of the other pretreatment systems satisfy a majority of the above criteria, and will not be considered further.

Acid Hydrolysis

The major criteria for the selection of acid hydrolysis processes are:

- Yields;
- Sugar concentrations;
- Hemicellulose degradation;
- Pilot-scale demonstrations;
- Capital and operating costs.

In all cases, wood chip pretreatment by explosive decompression is considered essential if high yields and high sugar concentrations are to be achieved.

Multi-stage dilute acid hydrolysis processes, applied after autohydrolysis, appear to have the potential of meeting the first two criteria. The process scheme, tested in New Zealand, takes advantage of non-uniformity of cellulose by providing separate treatments for cellulose and hemicellulose and should meet the third criteria. Capital and operating costs will depend on whether continuous operation is possible at high pulp consistency and equipment tests should be conducted. This technology appears to have the greatest long-term potential and has therefore been recommended.

Strong acid processes, after tests on laboratory and pilot plant scale, have been abandoned because of the economics. Some testing with strong acid processes should be possible on systems provided for two-stage dilute acid hydrolysis.

The solids recycle process should result in high yields. However, availability of suitable equipment is crucial. High sugar concentrations can only be achieved if very high yields are obtained per pass. Hemicellulose derived sugars must be extracted before hydrolysis to prevent degradation. Work to date indicates that yields per pass are low resulting in low sugar concentrations and high equipment costs as large solid volumes must be handled. This process is not favoured for application at the proposed facility.

Enzyme Production and Hydrolysis

The key to the success of enzyme hydrolysis process is enzyme production. In all cases, the enzyme is produced from microorganisms under anaerobic or aerobic conditions. Equipment can be provided at the proposed facility to accommodate enzyme production from these microorganisms.

The enzymes can be:

- produced on site, separated, concentrated and used, or
- produced on site and the broth used directly for hydrolysis.

To determine which of the above systems should be provided, a review of the practices established in starch ethanol plants is useful. In this case:

- Concentrated enzymes are supplied to small facilities by specialty enzyme producers. These enzymes are produced from a suitable substrate, separated and concentrated at supplier's location.

- Enzymes are produced on-site using plant substrate at large facilities, and the enzyme broth is used for direct hydrolysis by batch or continuous feeding of substrate to the broth, or vice versa.

As the economics of ethanol production from cellulose is expected to become favourable only for large-scale plants because of the high capital costs, the second method is expected to become more common. Some experience already points in this direction and the practice will be established quickly as enzyme production rates are improved by mutational and genetic manipulation techniques. Development work on the production of beta-glucosidase, which is a critical component of cellulose enzymes, and is not produced in sufficient quantities by Trichoderma viride, also continues, and should result in faster hydrolysis rates and higher yields. Therefore, on-site enzyme production and direct use of enzyme broth for hydrolysis is preferred. Space should, however, be provided for later installation of purification equipment if required for specific enzymes and microorganisms.

For enzyme hydrolysis, batch or continuous reaction in stirred tank reactors is most appropriate. This system will also accommodate simultaneous saccharification fermentation (SSF) or direct production of ethanol from cellulose by bacterial action. Plug flow reactors, although of interest, will not affect the economics significantly and should not be considered further.

Simultaneous saccharification fermentation (SSF) is based on the theory that high sugar concentrations retard enzymatic activity. This process has been tested on a pilot plant scale and further progress is reported. It, however, compromises optimum conditions for both hydrolysis and fermentation. Once enzymes with suitable activity levels, including beta-glucosidase are developed, a series hydrolysis fermentation may be the best answer. However, SSF should be considered further as an alternative enzymatic hydrolysis process.

Processes, which favour contact of hydrolyzate with fresh substrate to enhance enzyme utilization, are not recommended because of the high cost of handling equipment. Improvements in enzyme activity should be the long term objective.

Production of cellulase by thermophilic bacteria is in the initial stages of development. This process should be monitored for future testing on a bench-scale.

## Alternative Process Configurations

Based on the technologies selected, a number of alternative process configurations have been formulated. Figures 1, 2 and 3 present these configurations. Pretreatment by steam explosion, distillation and by-products from pentose sugars are common to all options. Distinctive features of each option are:

Alternative 1: Enzymatic Hydrolysis, Lignin Separation and Fermentation

Alternative 2: Lignin and Hemicellulose Separation, Enzymatic Hydrolysis and Fermentation

Alternative 3: SSF, Lignin Separation

Alternative 4: Lignin and Hemicellulose Separation, SSF

Alternative 5: Acid Hydrolysis, Lignin Separation, Fermentation.

Alternative 6: Lignin and Hemicellulose Separation, Acid Hydrolysis, Fermentation.

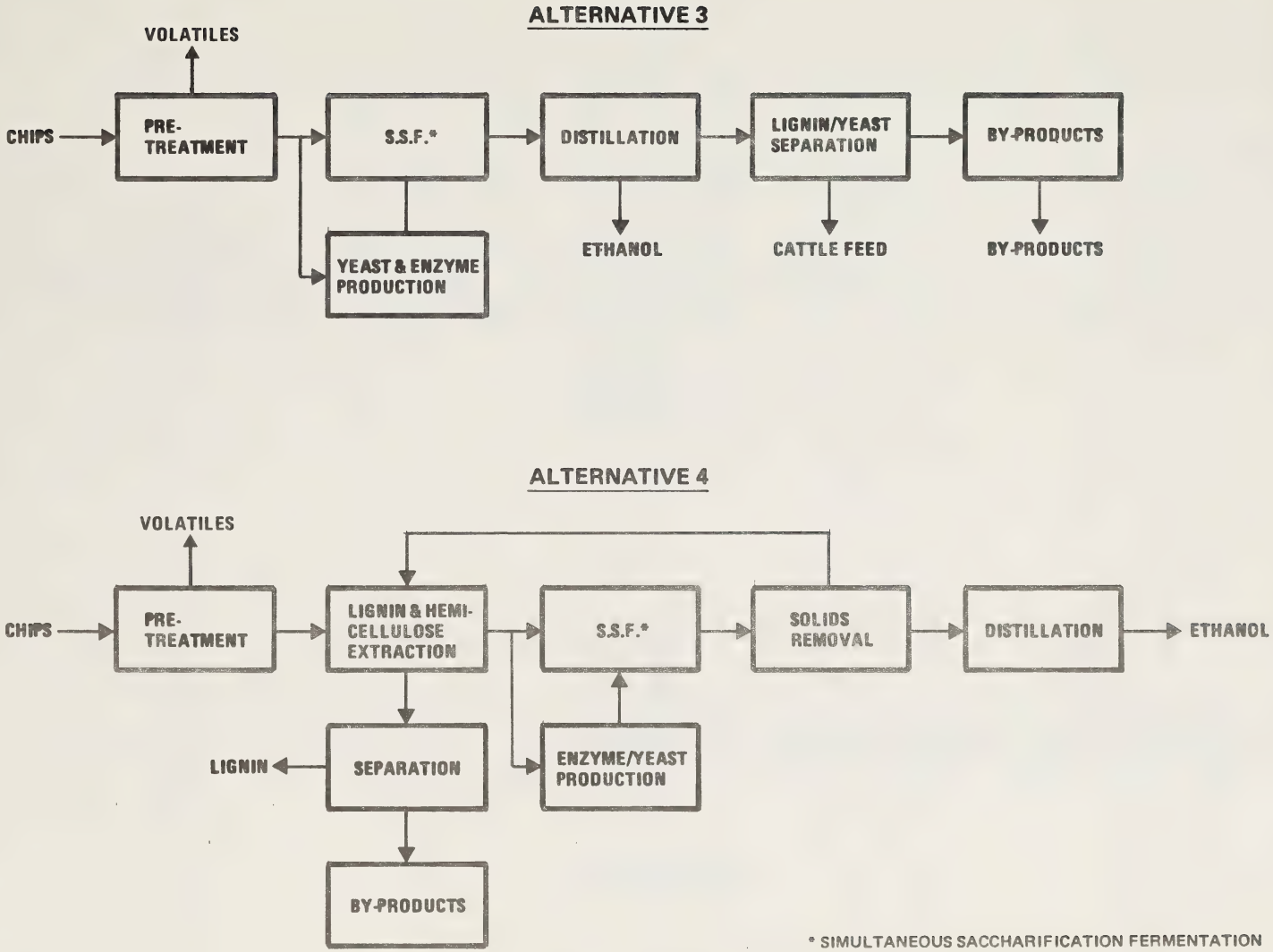
### Alternative 1

This alternative involves separation of lignin after enzymatic hydrolysis, and production of by-products from pentose sugars present in the stillage. It represents perhaps the simplest and most logical route to ethanol from lignocellulosics and has been tested with some success by Iotech Corporation at Gulf's pilot plant in Kansas. A major problem is the imbalance in enzyme activity caused by a lack of beta-glusocidase. Research work continues on the production of this cellulose component. Additional pilot and demonstration work is required to confirm yields, sugar concentrations, reaction times and economics.

Lignin recovered by this route contains mycelia, and further purification may be required to achieve a high quality product.

ALTERNATIVE ENZYMATIC HYDROLYSIS CONFIGURATIONS

FIGURE 1




ALTERNATIVE ENZYMATIC HYDROLYSIS CONFIGURATIONS (S.S.F.) FIGURE 2

ALTERNATIVE PROCESS CONFIGURATIONS - ACID HYDROLYSIS FIGURE 3

Alternative 2

In this case, lignin and hemicellulose are removed before hydrolysis. A major advantage is the high quality of lignin thus obtained. However, complete lignin recovery may not be realistic in a single stage operation and separation following hydrolysis may become necessary.

Alternative 3

This alternative involves simultaneous saccharification fermentation (SSF). Lignin is removed following distillation along with yeast and mycelia and used as cattle feed. One of the drawbacks of this process is the high cost of yeast production, as fresh yeast must be produced for each batch.

Alternative 4

This involves pre-separation of lignin and hemicelluloses, and simultaneous saccharification fermentation. The merits and disadvantages of this process are similar to those defined for Alternative 2.

Alternative 5

This process is similar to Alternative 1. However, lignin thus obtained is suitable for use as a fuel only. Some hemicellulose degradation is also expected, resulting in a lower by-product recovery.

Alternative 6

This process will recover high quality lignin and hemicellulose. However, part of the lignin recovered after hydrolysis will be useful as a fuel only.

## Selection of Process Configurations

The Terms of Reference require that the proposed facility provide flexibility to test a number of process configurations. Further configurations or technologies may be tested by modifying installed equipment and systems or adding new equipment.

However, it may not be possible to test all of the various configurations with equal ease on a continuous basis. Capacities may also vary for each case. To provide a focus for the design and operation of the facility, the following approach is proposed:

a) Select primary processes which best meet the selection criteria. Design the plant to meet specified capacities and continuous operation requirements for these processes.

b) Select secondary processes for which the main process streams can be tested on a continuous basis, but lower or higher capacities may be achieved. Some of the by-product systems may not operate parallel to the main process stream.

c) Determine what additional processes can be tested with or without minor modifications.

d) Provide flexibility in design to incorporate additional systems and equipment at a later date.

## Selection Criteria

The following selection criteria are defined in descending order of significance:

1. Capability to produce ethanol with a high emphasis on by-products.

2. Process developed preferably to a pilot-scale for poplar: as the objective of the proposed facility is to demonstrate the technical and economic feasibility of the various processes, and to provide scale-up data, it is desired that some pilot-scale test data be available.

3. Flexibility to accommodate improvements: Improvements in cellulose conversion technologies are expected in the following areas:

   - More energy efficient and effecitve pretreatment.
   - Higher enzyme activity and low enzyme production costs.
   - Higher sugar concentrations (greater than 12%).
   - Incorporation of pentose fermentation to ethanol.
   - High quality lignin recovery.
   - Reduced energy consumption.

4. Economic potential and capital cost: Although overall economics are the key, low capital costs will help establish the industry.

5. Availability of design data: It is desirable that adequate design data be available for optimum design of the facility and assessment of costs.

6. Proprietary status: The proprietary status should be clear so that negotiations can be carried out with the process vendors.

Based on these criteria, specific process configurations have been recommended for the enzymatic and acid hydrolysis processes.

## Enzymatic Hydrolysis Process Selection

The following four alternative process sequences were considered for enzymatic hydrolysis:

a) Alternative 1 - Sequential hydrolysis and fermentation with intermediate lignin separation.

| CRITERIA \ PROCESS ALTERNATIVES | ALTERNATIVE 1<br>HYDROLYSIS, LIGNIN SEPARATION, FERMENTATION | ALTERNATIVE 2<br>LIGNIN SEPARATION, HYDROLYSIS, FERMENTATION | ALTERNATIVE 3<br>* SSF, DISTILLATION, LIGNIN SEPARATION | ALTERNATIVE 4<br>* LIGNIN SEPARATION, SSF |
|---|---|---|---|---|
| 1. CONVERSION WITH EMPHASIS ON BY-PRODUCTS | HIGH EMPHASIS | HIGH EMPHASIS | LOW | HIGH EMPHASIS |
| 2. BASIC DEMONSTRATION ON A PILOT-SCALE FOR POPLAR. | LIMITED DEMONSTRATION | NOT DEMONSTRATED | DEMONSTRATED WITH LIMITED SUCCESS FOR MUNICIPAL WASTES | NOT DEMONSTRATED |
| 3. FLEXIBILITY TO ACCOMODATE IMPROVEMENTS | EXCELLENT | EXCELLENT | GOOD | GOOD |
| 4. ECONOMIC POTENTIAL AND CAPITAL COST | EXCELLENT OVERALL ECONOMIC POTENTIAL | GOOD POTENTIAL BUT DEPENDS ON LIGNIN VALUE | FAIR POTENTIAL | GOOD POTENTIAL BUT DEPENDS ON LIGNIN VALUE |
| 5. AVAILABILITY OF DESIGN DATA | SOME DATA AVAILABLE | SOME DATA AVAILABLE | SOME DATA AVAILABLE | SOME DATA AVAILABLE |
| 6. PROPRIETARY STATUS | NOT YET CONFIRMED; IOTECH CLAIMS PATENTS PENDING | PARTIALLY OWNED BY A GERMAN FIRM; STAKE DISPUTES SUCH OWNERSHIP | GULF OWNS PATENTS ON SSF | GULF OWNS PATENTS ON SSF. LIGNIN SEPARATION PATENT BY A GERMAN FIRM, DISPUTED BY STAKE |

* SSF - SIMULTANEOUS SACCHARIFICATION FERMENTATION

**ENZYMATIC HYDROLYSIS**
PROCESS CONFIGURATION SELECTION

**TABLE**

b) Alternative 2 - Sequential hydrolysis and fermentation; lignin separation before hydrolysis.

c) Alternative 3 - Simultaneous saccharification-fermentation (SSF); lignin separation after distillation along with yeast.

d) Alternative 4 - SSF with lignin separation before fermentation.

Table 3 presents an evaluation of these alternatives and a discussion of their merits and disadvantages follows:

1. Alternative 1 appears to be the most attractive process from the standpoint of by-products recovery. Its basic components of hydrolysis, lignin separation and fermentation have been tested by Iotech at Gulf's pilot plant on exploded wood substrate, although improvements in enzyme activity are required to reduce costs. Sugar concentrations as high as 12% were obtained. The process can easily incorporate improvements in ethanol technology. It is the most direct method of making ethanol from wood, therefore, capital costs are expected to be lower than the other processes. Iotech claims to have patent applications pending and its proprietary status is not very clear.

2. Alternative 2 has not been demonstrated on a pilot scale. Its strength lies in the quality of lignin extracted. However, the capital and operating costs for such extraction are high. Ownership of technology relating to lignin and hemicellulose appear to be in dispute, although a German firm has a patent in Canada.

3. Alternative 3 extracts lignin as low quality cattle feed. Attempts to demonstrate this process with municipal wastes by Gulf were not very successful, and the process has not been demonstrated on wood. However, drastic improvements in pretratment and enzyme activity have recently been claimed. Patents on SSF are owned by Gulf Oil and Chemical Company, and are marketed internationally by Raphael Katzen Associates, Inc.

4. Alternative 4 is similar to Alternative 2, except that SSF is employed for hydrolysis and fermentation.

The following conclusions are drawn from the above analysis:

a) Alternative 1 is the preferred primary process configuration, and the plant system under continuous operation should be based on this scheme.

b) Alternative 2 is the preferred secondary process configuration.

c) Alternative 4 closely matches Alternative 2 and its testing, with minor modifications to the equipment and systems, should be possible. These modifications will generally be required in the enzyme production, hydrolysis and fermentation units.

Acid Hydrolysis Process Selection

The following acid hydrolysis process configurations were considered:

Alternative 5. Lignin separation after acid hydrolysis.
Alternative 6. Lignin extraction before acid hydrolysis.

As the two configurations are essentially the same as selected for the primary and secondary enzymatic hydrolysis, both can be provided by the addition of an acid hydrolysis system.

However, Alternative 6 is preferred as the primary process as it recovers a high quality lignin by-product.

## TECHNOLOGY OWNERSHIP AND STATUS

Most of the technologies being developed for ethanol production from lignocellulosic materials are based on proprietary processes so that design information is not readily available. Cooperation of process suppliers may not always be forthcoming. Also, in some cases, ownership of technology is disputed. Efforts were made to clarify these points and included the following steps:

- Site visits to: Gulf's pilot plant at Pittsburgh, Kansas; Georgia Institute of Techonology (GIT) in Atlanta; Wenger Manufacturing in Sabetha, Kansas; Iotech, Stake Technology Ltd., and National Research Council (NRC) Laboratories in Ottawa; Masonite Canada Ltd. and Canadian International Paper Ltd. in Gatineau, Quebec; and Dr. Laszlo Paszner at the University of British Columbia. A seminar presented by Dr. Derrik Whitworth in Ottawa on New Zealand's acid hydrolyis process was also attended.

- A Canadian patent search to determine ownership status of various technologies.

- Process tests on Wenger equipment using Alberta hardwoods; enzymatic hydrolysis of exploded wood; and pumping of high consistency cellulose slurries using an open-throat Moyno pump.

### Site Visits

Appendix B contains detailed site visit reports by Prof. Morris Wayman and Dr. Hidayat Husain. Major findings are discussed below:

## Gulf Pilot Plant

This pilot plant, located at Gulf's Pittsburgh, Kansas, petrochemical complex, was visited and discussions held with plant operators. It was determined that:

- Serious contamination problems with municipal wastes prevented successful testing of SSF process.

- Iotech had some success with enzymatic hydrolysis of exploded wood. Ethanol concentrations up to 6% were achieved, although reproducibility was poor because of the difficulty in maintaining enzyme quality and activity. Microorganisms were brought in from other research centers, and their characteristics varied from batch to batch.

## Iotech Corporation

Discussions were held with Mr. Geoffrey Noble, Vice President, R&D, Iotech Corporation in Ottawa regarding technology ownership, potential cooperation and possible Iotech involvement in the proposed facility. Salient points are as follows:

- Iotech claimed that Canadian patents are pending on both their autohydrolysis process and their process scheme tested at Gulf's pilot plant.

- Iotech will negotiate licensing their technology to the proposed facility at a suitable time.

- Iotech will use the proposed demonstration plant for their process development only if they have complete control of operations and information transfer during these tests.

- They recommend a 25 tonne/day BDWE using a rapid cycle autohydrolyzer for the proposed facility.

Stake Technology Ltd.

In a meeting with Stake Technology Ltd.'s Vice President, Mr. Doug Brown, he made the following points:

- Stake will prefer to sell a complete system including autohydrolyzer, hemicellulose and lignin extraction system, and acid hydrolysis equipment. He claimed that patents were pending for this process.

- Stake will negotiate technology licensing with Alberta Energy and Natural Resources during the design phase.

- He recommended a minimum of 2.5 tonnes/day BDWE capacity for testing Stake's system and process.

- Stake is interested in using the demonstration plant if it is available at suitable terms.

NRC Laboratories, Ottawa

A meeting was held with Dr. Henry Schneider, Mr. John Vose and Dr. Claude Bishop of NRC regarding the status of pentose technology. Information obtained during this meeting has been discussed in the Technology Review Section.

University of British Columbia

Dr. Laszlo Paszner's organosolv saccharification process was discussed with the inventor during this visit. Details of the process have been discussed in the Technology Review Section. Dr. Paszner indicated a willingness to participate with Alberta Energy and Natural Resources on the proposed project at suitable terms to be negotiated at a later date.

Wenger Manufacturing Inc.

Wenger's Sabetha, Kansas, manufacturing facility was visited to observe tests on Ontario poplar and to discuss the application of their equipment at the proposed facility.

- Tests with poplar appeared successful, although chips had to be ground to facilitate feeding. They believe they can feed chips to a larger unit without problems.

- Wenger is an equipment manufacturer and not a process supplier. They normally obtain only defensive patents and are prepared to sell their equipment without licensing agreements.

- They agreed to run tests on Alberta hardwood if sufficient quantities were made available.

Other Visits

Visits to Georgia Institute of Techology (GIT), Masonite Canada Ltd., and Canadian International Paper's Gatineau facilities were mainly aimed at obtaining design data on autohydrolysis, acid hydrolysis and continuous fermentation. Most of the information thus obtained was used for conceptual design and costing activities.

A seminar on New Zealand's research in acid hydrolysis, presented by Dr. Derrik Whitworth in Ottawa, was also attended, and a detailed report is included in Appendix C.

Patent Search

A Canadian patent search was carried out through the firm of Hirons, Rogers & Scott. Their letter outlining the results of the search is attached in Appendix D. Following conclusions are drawn:

1. The most important patent which covers autohydrolysis in the range of 160-230°C for 2 - 480 minutes, alkaline extraction and enzymatic or acid hydrolysis is held by Dietrichs. This Stake type autohydrolysis is covered if coupled with alkaline extraction, and is very similar to the secondary process proposed for the demonstration plant. Iotech and Wenger autohydrolysis processes are not covered by this patent.

2. Two patents are issued to Stake, but these cover the autohydrolyzer feed system.

3. No patents are assigned to Iotech.

4. DeLong has a patent on a batch autohydrolysis process similar to Iotech's, and this patent has been assigned to Canada Patents and Development Ltd., a Crown corporation.

5. No patents are issued to Gulf Canada.

Process and Equipment Test

Following process and equipment tests were commissioned during the study:

- Autohydrolysis of Alberta's poplar using Wenger extruder. The extruded wood was then hydrolyzed in Professor Wayman's laboratories using commercial enzymes.

- Pumping of high consistency pulp slurries using an open-throat Moyno pump.

Correspondence, outlining the results of these tests, is attached in Appendix E. In both cases, the tests were highly successful. The Wenger extruder was determined to be quite satisfactory for the treatment of poplar wood. Up to 30 percent solids slurry could be pump by open-throat Moyno with negligible slippage. Only 10 percent slippage occurred at 40 percent consistency.

RESEARCH AND DEVELOPMENT

## Introduction

A review of the existing technologies indicates that a comprehensive research and development program is required to commercialize processes for making ethanol from wood. These comprehensive research needs are presented in Appendix F and cannot be fulfilled by a single facility.

However, some basic research on specific processes and products should be performed under this program, and the scope of such research is outlined in the following section. Approaches for the implementation of an R & D program are also presented.

## Research and Development Specific to Selected Processes

To achieve the objective yields and capacities and to improve the economic feasibility, research and development work is required for enzyme and acid hydrolysis processes and for the conversion of lignin to high value secondary products. This work can be carried out parallel to the design, construction and operation of the pilot plant.

Research is also required in the fermentation, distillation and the integrated fermentation/distillation operations. However, this is being performed by universities and industry for application in the conventional sugar and grain based ethanol industries. These efforts need not be repeated at this facility to maintain the focus on hydrolysis.

## Basic Research

This should include the following:

- Microorganism selection and mutation for improved enzyme production. It is probable that a number of microorganisms with cellulase, beta-glucosidase, and hemicellulase producing capabilities will be required in addition to Trichoderma viride.

- Microbe selection, and genetic engineering for hexose and pentose fermentation.

- Lignin chemistry, purification and conversion to develop high value products.

- Corrosion testing of metals under hot sulphuric acid environment.

- Kinetics and reaction mechanism for enzymatic and acid hydrolysis.

- Chemistry of volatile by-products of autohydrolysis and acid hydrolysis.

- Separation of by-products present in the stillage.

Bench Scale Work

- Development of the organosolv saccharification process developed by Dr. Laszlo Paszner at the University of British Columbia.

- Development of processes for enzyme production using low cost nutrients; enzyme separation and recycle; immobilized enzymes for cellulose hydrolysis and xylose isomerization to produce ethanol using Saccharomyces cerevisiae.

- Optimization of a multi-stage acid hydrolysis process.

- Lignin purification and process development for high value derivatives.

- Processes for recovery of valuables from process wastewater and volatiles.

## Pilot Scale Work

Process development work on the pilot scale should include:

- Optimization of pretreatment processes to minimize hemicellulose degradation.

- Use of a catalyst during autohydrolysis to achieve partial or complete cellulose hydrolysis.

- Development of effective methods of feeding substrate to the acid hydrolysis reactors.

- Improvements in solid-liquid separation procedures to increase sugar concentrations.

- Development of methods for energy recovery and air pollution reduction from autohydrolysis and acid hydrolysis processes.

## Equipment Design, Development and Testing

One of the major challenges at the demonstration plant will be the conveying, mixing, pumping and dewatering of high consistency pulps. Work on testing and development of equipment for these processes will be desirable. The objective is to achieve maximum sugar recovery at high sugar concentrations. Some equipment testing may be desirable before the design of the demonstration plant to select systems which will help in achieving plant objectives.

Design of reactors for enzymatic and acid hydrolysis also requires attention.

R & D Program Implementation

The basic objective of the proposed program is to speed up the development of wood to ethanol technologies in order to improve the feasiblity of Alberta's polar resource utilization. To achieve this objective, it is proposed that:

a) Alberta should take a lead in developing and implementing a comprehensive R & D program. This program should involve: basic and applied research specific to develop preferred processes to a point where these can be demonstrated at the proposed facility, and products utilization. New processes with a high potential should also be included. Technology ownership and licensing policies similar to those adopted by Alterta Oil Sands Technology Research Authority could be used for this R & D work.

b) Private enterprise should be encouraged to participate in the above program generally, and especially in using the demonstration plant by creating a conducive environment, through appropriate management structure and policies.

Potential options for the implementation of R & D are:

1. All laboratory, bench-scale and pilot-scale research and development work to be performed at the proposed facility.

2. All work to be contracted out to universities, research centres and private firms, and to be performed at contractor's facilities.

3. Some basic work to be contracted out; bench and pilot-scale work to be carried out by the plant staff.

4. All R & D work to be contracted out; however, bench and pilot-scale work to be performed by the contractor at the demonstration plant with assistance from the plant staff.

Option 1 will require an extensive research laboratory facility at the demonstration plant. Cost of such a laboratory is expected at $12 - $15 million. Manpower requirements are estimated at 80 - 100, and the annual operating budget at $4 - $5 Million. Because of involvement of plant personnel in in-house R & D, private firms may not use the facility to protect their proprietary technology. In view of the high costs and conditions unfavourable to the private sector, this option is not recommended.

Option 2 has the advantage that it develops expertise at universities and other research centres. It also encourages participation by private enterprise. Technology ownership and secrecy questions can be easily addressed. However, it assumes that suitable bench and pilot-scale facilities are available for process development elsewhere. This is, of course, not valid, as the lack of suitable pilot and demonstration plants is one of the major hurdles in commercialization of wood to ethanol technologies. Construction of separate pilot-scale facilities for each location would also be expensive and time consuming. Therefore this alternative is not recommended.

The third alternative would involve provision of analytical capabilities, and bench-scale testing facilities at the demonstration plant laboratory. Estimated cost of the laboratory equipment is $1.23 Million. This alternative optimizes the use of expertise available at the plant, while developing expertise and manpower at the universities by funding basic research. However, the potential conflict of interest between the organization and the private enterprise remains and this alternative is therefore not favoured.

The fourth option requires similar lab facilities to the third alternative, and has the same advantages as Option 3. However, it offers better protection of the process owner's technology by limiting the role of the pilot plant staff to only assisting contractors for all R & D work. Separation of the operation and R & D function should encourage participation by private enterprise in this program. This option is recommended for the management of the R & D program.

Conclusions

The key to commercializatin of poplar-ethanol technologies is the development of hydrolysis processes and by-products from lignin. Research and development activities should focus on these areas.

It is proposed that all basic and applied research and development work be contracted out to Universities and other research centres. However, the unique facilities and expertise at the proposed plant can be used by these contractors at suitable stages of process development.

PLANT CONCEPT

## Introduction

The Terms of Reference required that flexibility to test the maximum number of technologies be provided. This criterion formed one of the basic guidelines during the System Selection phase. It is also preferred that the processes be demonstrated under continuous operation, and that such operation should provide scale-up data for a commercial scale facility. These objectives must form the basis of plant concept development, selection of equipment, and of the process instrumentation and control philosophy.

## Capacity

A plant capacity in the range of 1 t/d of BDWE to 50 t/d of BDWE was specified in the Terms of Reference. Other objectives, which influence plant capacity selection, are:

- Demonstration under continuous operation.
- Development of scale-up data.
- Capital and operating costs.

Discussions with process developers and equipment manufacturers, and in particular pulp handling and dewatering equipment suppliers, lead us to conclude that a minimum of 10 tonnes/day BDWE capacity is essential to achieve the first two objectives. The data obtained at the plant will have to be scaled-up by factor of 25 to 100 for commercial scale plants. This is an acceptable figure for the chemical industry. Pretreatment system suppliers recommended a 25 t/d BDWE capacity. On this basis:

- Primary plant capacity was selected at 10 t/d BDWE with pretreatment systems sized at 25 t/d BDWE.

- Alternate plant capacity was selected at 25 t/d BDWE, and costs were determined for this case to assess the variation of cost with size.

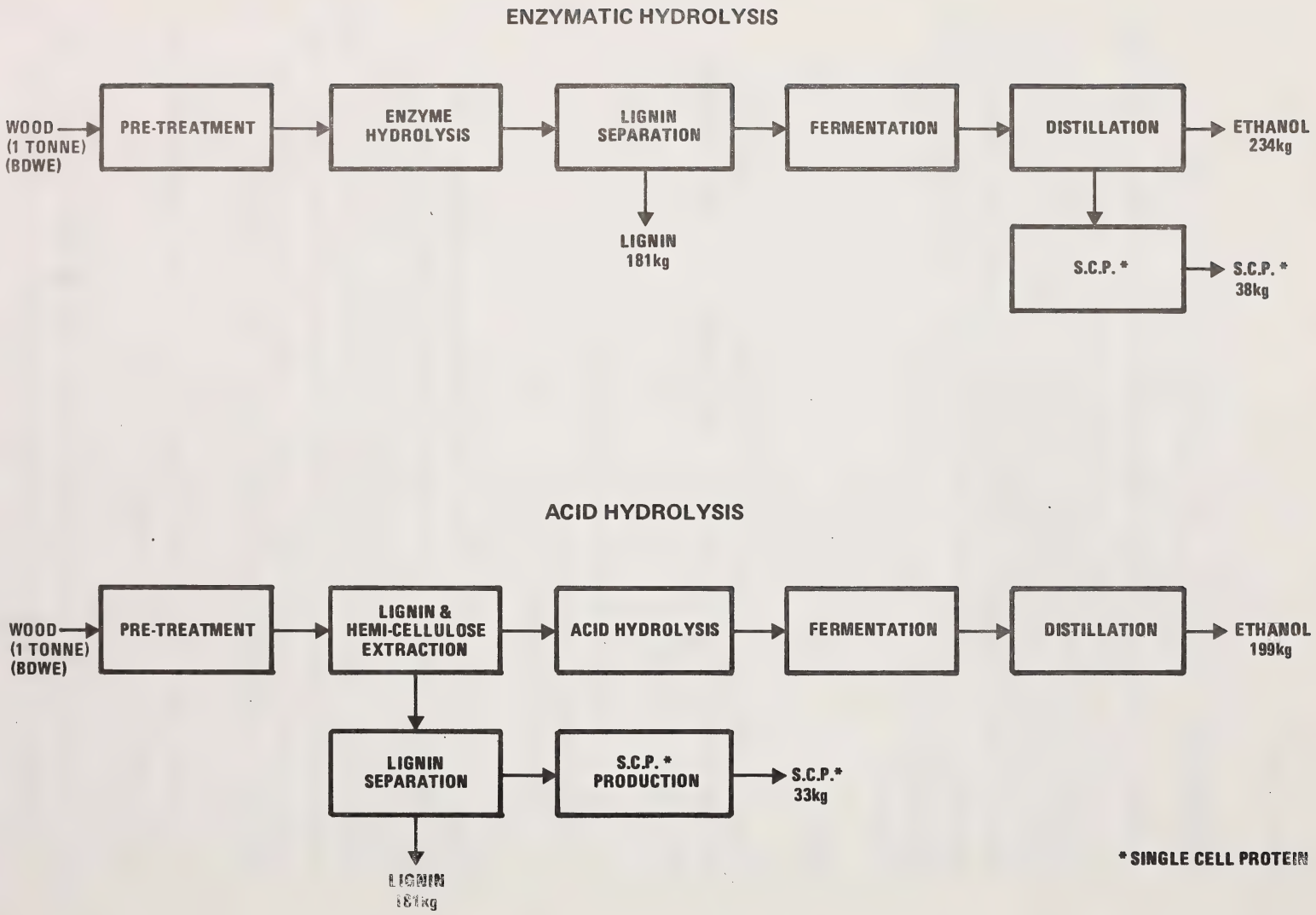

ENZYMATIC HYDROLYSIS
WOOD (1 TONNE) (BDWE)
PRE-TREATMENT
ENZYME HYDROLYSIS
LIGNIN SEPARATION
FERMENTATION
DISTILLATION
ETHANOL 234kg
LIGNIN 181kg
S.C.P. *
S.C.P. * 38kg
ACID HYDROLYSIS
WOOD (1 TONNE) (BDWE)
PRE-TREATMENT
LIGNIN & HEMI-CELLULOSE EXTRACTION
ACID HYDROLYSIS
FERMENTATION
DISTILLATION
ETHANOL 199kg
LIGNIN SEPARATION
S.C.P. * PRODUCTION
S.C.P.* 33kg
LIGNIN 181kg
* SINGLE CELL PROTEIN

FIGURE 4

Material Balances

Figures 4 and 5 define material balances for the following process configurations:

- Figure 4: Primary Enzymatic and Acid Hydrolysis Processes
- Figure 5: Secondary Enzymatic Hydrolysis Process

These balances are based on yields and conditions which should be the objectives for the development of these processes.

Overall assumed conversion efficiencies, based on cellulose, are as follows:

1. Primary enzymatic = 81%
2. Secondary enzymatic = 83%
3. Primary Acid = 69%

Lower overall conversion is assumed for the primary enzymatic process than the secondary enzymatic process because of sugar losses with lignin. These losses can be reduced but will result in dilution of sugar solution. In actual practice, sugar recovery and concentrations will have to be optimized from an economic standpoint.

The above figures include efficiencies of pretreatment, hydrolysis, fermentation and distillation. Whereas fermentation, distillation and pretreatment efficiencies are based on experience and calculated performance of pulp handling equipment, the following conversion factors have been assumed for hydrolysys:

| | | |
|---|---|---|
| Enzymatic hydrolysis | = | 95% |
| Acid hydrolysis | = | 80% |

The 95 percent enzymatic hydrolysis efficiency results from the use of 5 percent of the substrate for enzyme production. Hydrolysis efficiency should approach 100%.

SECONDARY PROCESS - MATERIAL BALANCE

FIGURE 5

## Process Descriptions

The proposed processes for the pilot plant are divided into the eight sections listed below.

0100 Chip receiving and pretreatment.
0200 Enzyme production and hydrolysis.
0300 Lignin separation.
0400 Fermentation.
0500 Distillation
0600 Acid hydrolysis.
0700 Hemicellulose and lignin extraction.
0800 Single cell protein production (or pentose fermentation).

Process flow diagrams for these units are shown in Figures 6 to 13, and a diagram of the proposed steam system is presented in Figure 14.

In actual practice, these systems will be arranged to test primary, secondary and other potential process configurations. For these descriptions, only primary enzymatic and acid hydrolysis process configurations are considered.

### Chips Receiving and Pretreatment

Wood chips are received by truck, and are dumped into a hopper. Screw conveyers feed these through a screen to a chip receiving bin and finally to the storage bins. A magnetic separator installed between the chip receiving bin and the storage bin removes the metallic debris. The Masonite gun, Stake reactor, and Wenger extruder are employed for the chip pretreatment. Only one reactor is operated at any time. The wood chips from the storage bin are transported through a screen into the feed tank of the reactor under operation. Oversize chips are rejected by the screen and are rechipped in a chipper.

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ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY
CHIP RECEIVING AND PRE-TREATMENT - 0100
817028-01-00
0
SEPT 1981
N.T.S
V.J. KEWITT
SN-0101
CV-0108
CV-0109
CV-0107
CV-0106
CV-0110
T-0107
T-0106
T-0105
T-0104
T-0103
T-0102
FD-0107
FD-0106
FD-0105
FD-0104
FD-0103
FD-0102
WENGER EXTRUDER R-0103
STAKE REACTOR R-0102
TIMER
STEAM
MASONITE GUN R-0101
CY-0103
CY-0102
CY-0101
CYCLONE SEPARATOR
SURGE TANK
T-0108
SCREW CONVEYOR CV-0201 FROM ENZYME PRODUCTION AND HYDROLYSIS DWG. NO 817028-01-00
P-0103
P-0102
P-0101
E-0101
S-0101
SCRUBBER
CASES TO ATMOSPHERE
OVERFLOW
WATER



**FIGURE 6**

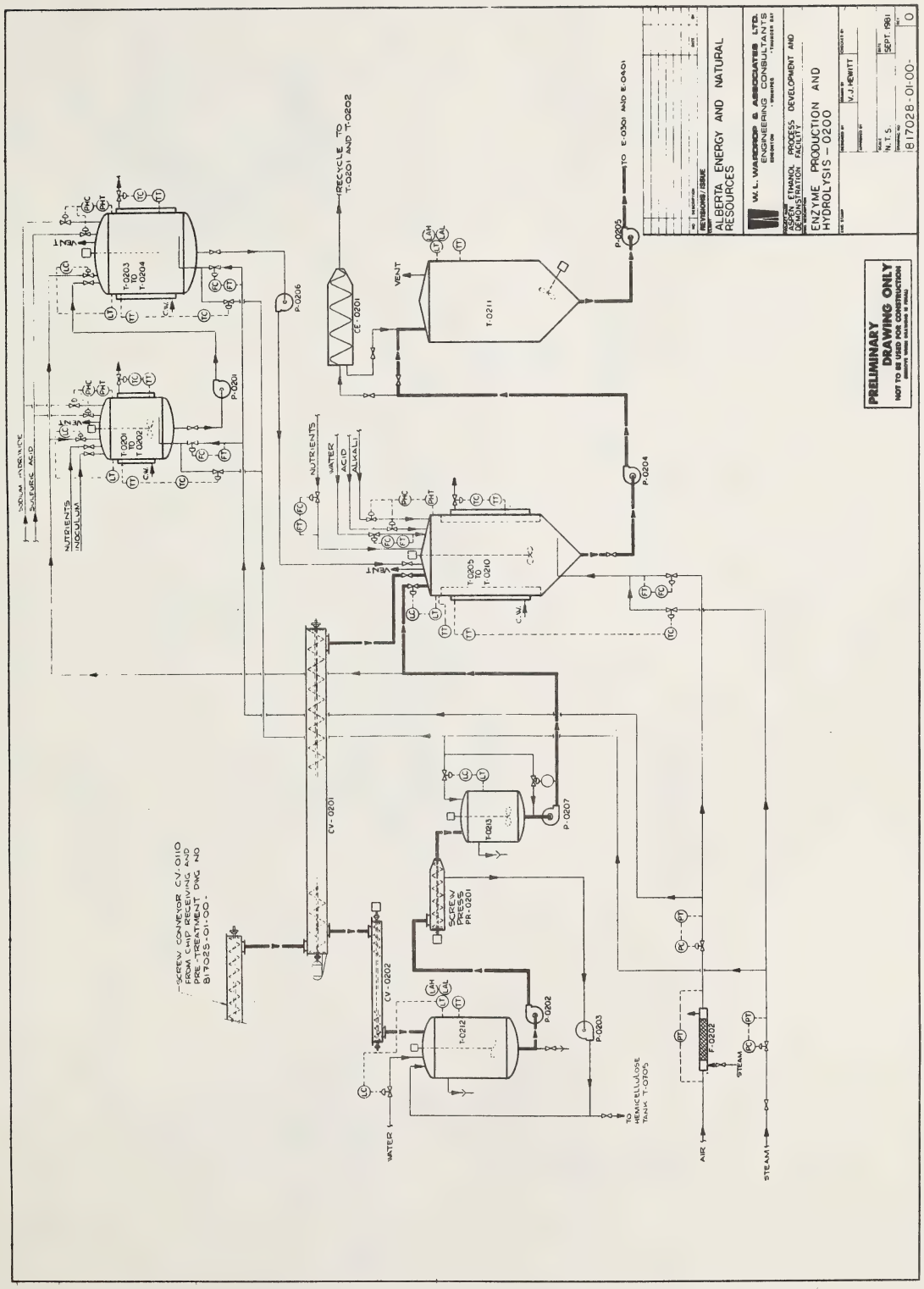
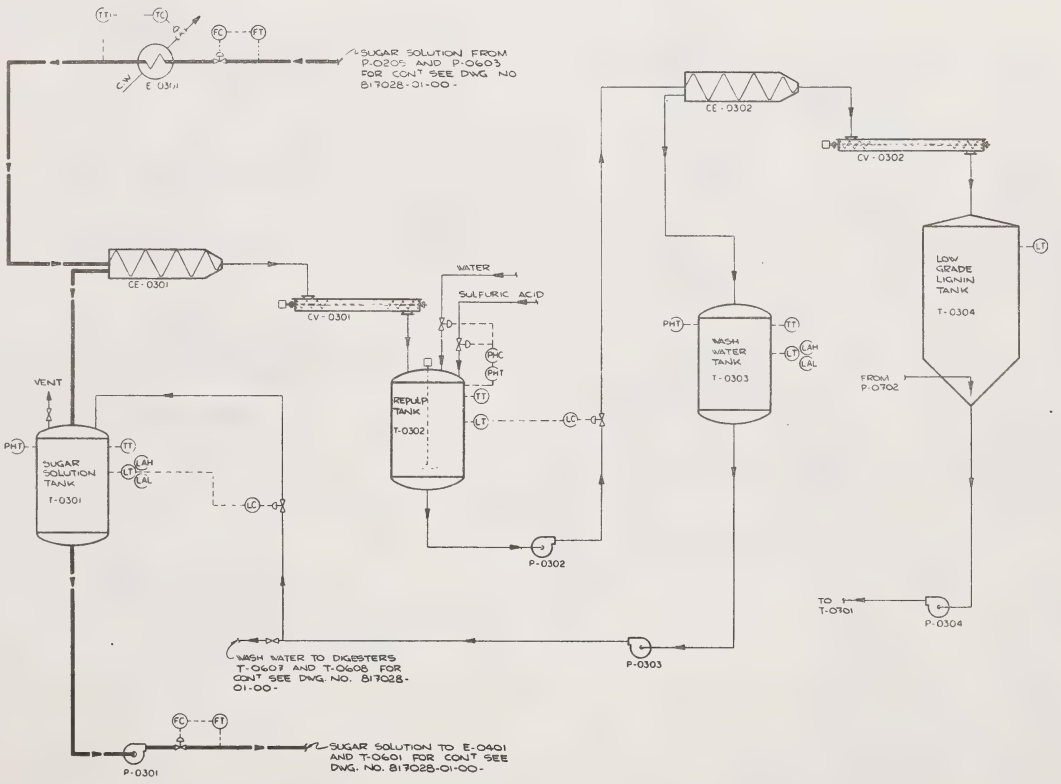

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ENZYME PRODUCTION AND HYDROLYSIS - 0200
817028-01-00-
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T-0203 TO T-0204
T-0201 TO T-0208
T-0209 TO T-0210
T-0211
T-0212
T-0213
CV-0201
CV-0202
PR-0201
P-0201
P-0202
P-0203
P-0204
P-0205
P-0206
P-0207
P-0208
CF-0201
RECYCLE TO T-0201 AND T-0202
TO E-0301 AND E-0401
SCREW CONVEYOR CV-0110 FROM HOPPER AND PRE-TREATMENT DWG NO 817025-01-00-
TO HEMICELLULOSE TANK T-0705
NUTRIENTS
INOCULUM
WATER
ACID
ALKALI
SODIUM HYDROXIDE
SULFURIC ACID
STEAM
AIR
VENT
C.W.



**FIGURE 7**




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PROJECT NAME: ASPEN ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY

DRAWING DESCRIPTION: LIGNIN SEPARATION - 0300

DESIGNED BY:
DRAWN BY: V.J. HEWITT
CHECKED BY:
APPROVED BY:
SCALE: N.T.S.
DATE: SEPT. 1981
DRAWING NO: 817028-01-00
REV: 0

FIGURE 8

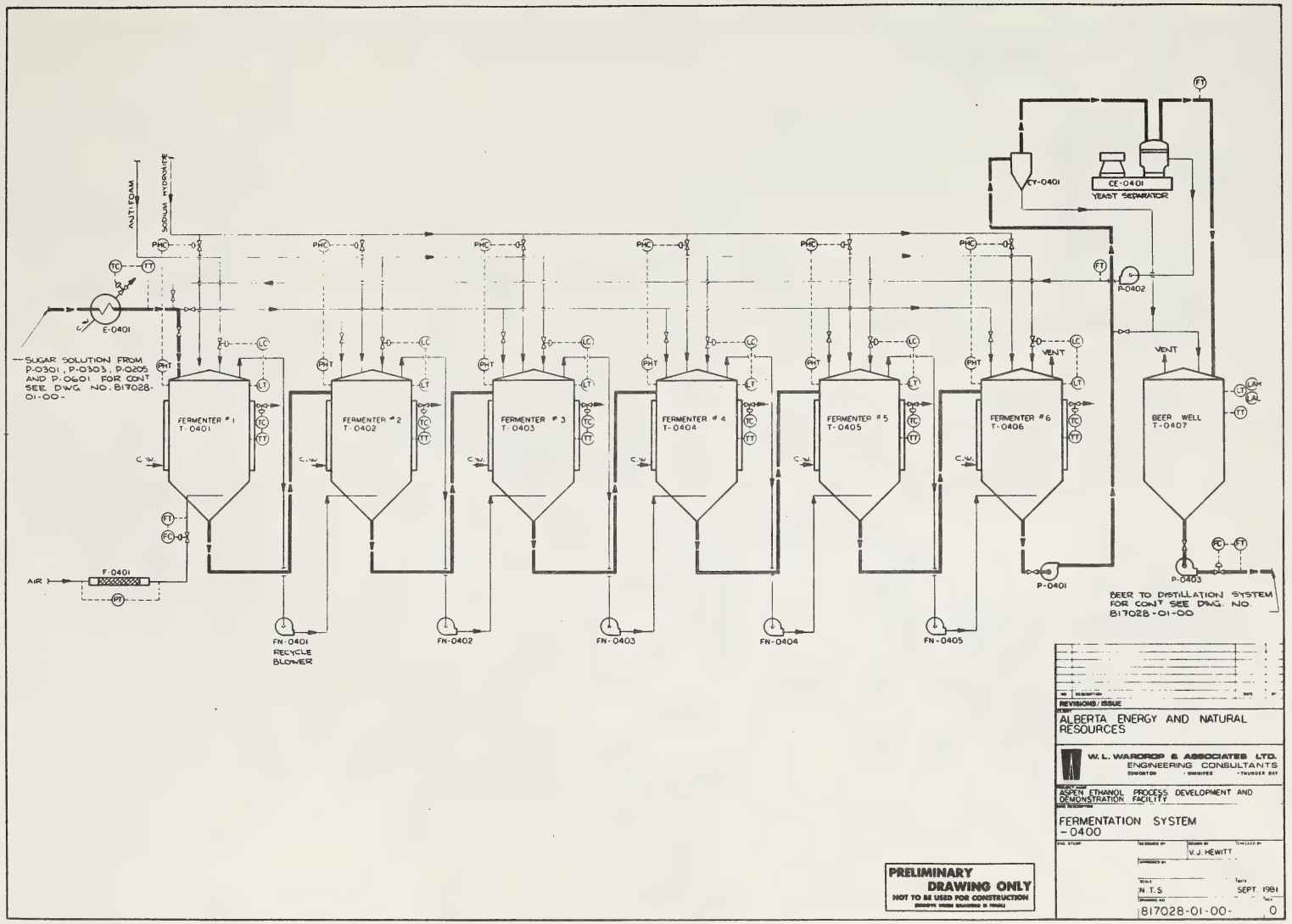

ANTI FOAM
SODIUM HYDROXIDE
E-0401
SUGAR SOLUTION FROM P-0301, P-0303, P-0205 AND P-0601 FOR CONT SEE DWG. NO. 817028-01-00-
FERMENTER # 1 T-0401
FERMENTER # 2 T-0402
FERMENTER # 3 T-0403
FERMENTER # 4 T-0404
FERMENTER # 5 T-0405
FERMENTER # 6 T-0406
BEER WELL T-0407
C.W.
AIR
F-0401
FN-0401 RECYCLE BLOWER
FN-0402
FN-0403
FN-0404
FN-0405
CY-0401
CE-0401 YEAST SEPARATOR
P-0402
P-0401
P-0403
VENT
BEER TO DISTILLATION SYSTEM FOR CONT SEE DWG. NO. 817028-01-00
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ASPEN ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY
FERMENTATION SYSTEM -0400
V. J. HEWITT
N. T. S
SEPT. 1981
817028-01-00-
0

FIGURE 9

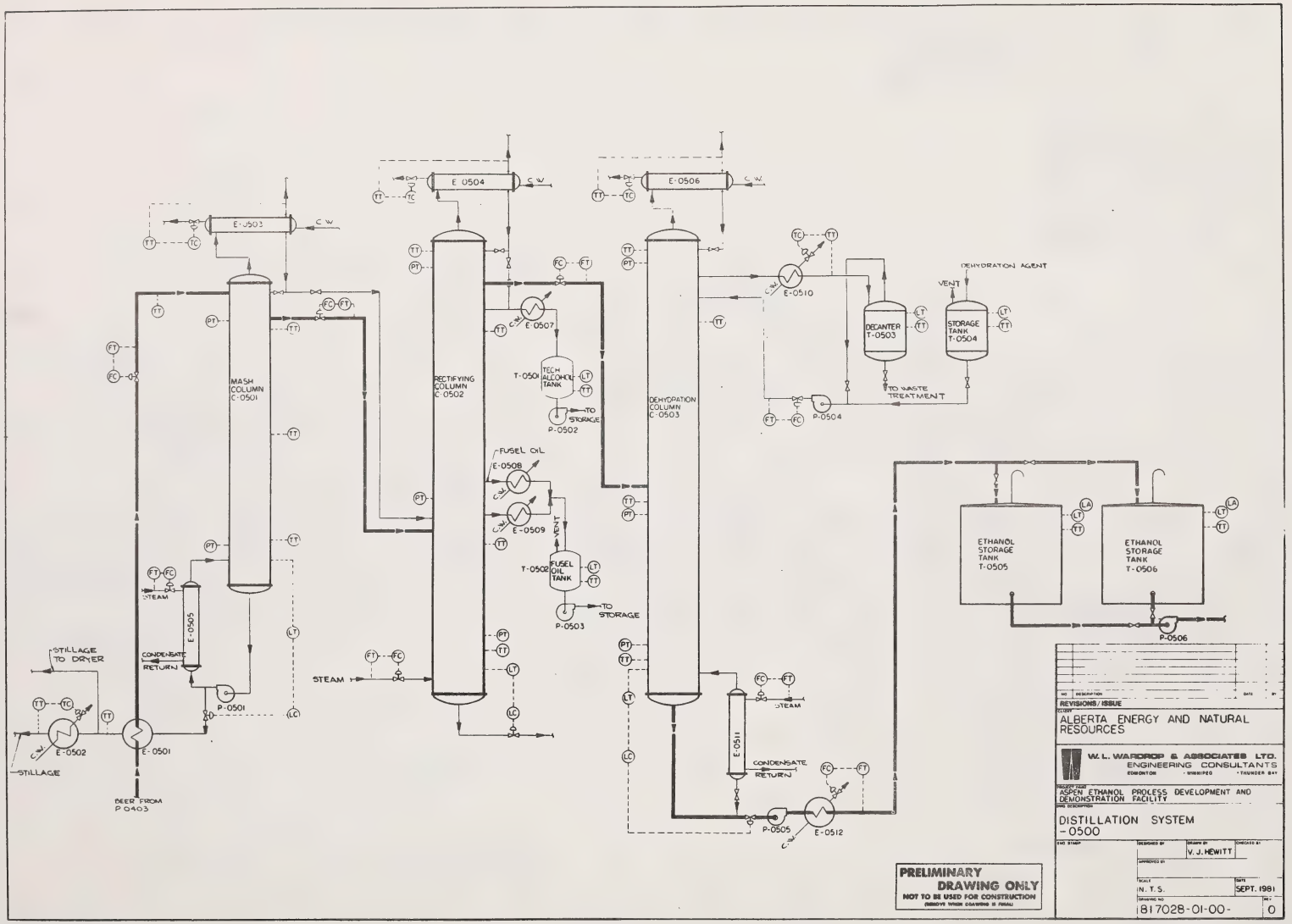

E-0503
E-0504
E-0506
C.W.
MASH COLUMN C-0501
RECTIFYING COLUMN C-0502
DEHYDRATION COLUMN C-0503
E-0507
E-0508
E-0509
E-0510
E-0511
E-0512
E-0501
E-0502
E-0505
T-0501 TECH ALCOHOL TANK
T-0502 FUSEL OIL TANK
T-0503 DECANTER
T-0504 STORAGE TANK
T-0505 ETHANOL STORAGE TANK
T-0506 ETHANOL STORAGE TANK
P-0501
P-0502
P-0503
P-0504
P-0505
P-0506
TO STORAGE
TO WASTE TREATMENT
DEHYDRATION AGENT
VENT
FUSEL OIL
STEAM
CONDENSATE RETURN
STILLAGE TO DRYER
STILLAGE
BEER FROM P 0403
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DISTILLATION SYSTEM -0500
DESIGNED BY
DRAWN BY V. J. HEWITT
CHECKED BY
APPROVED BY
SCALE N. T. S.
DATE SEPT. 1981
DRAWING NO 817028-01-00-
REV 0

FIGURE 10

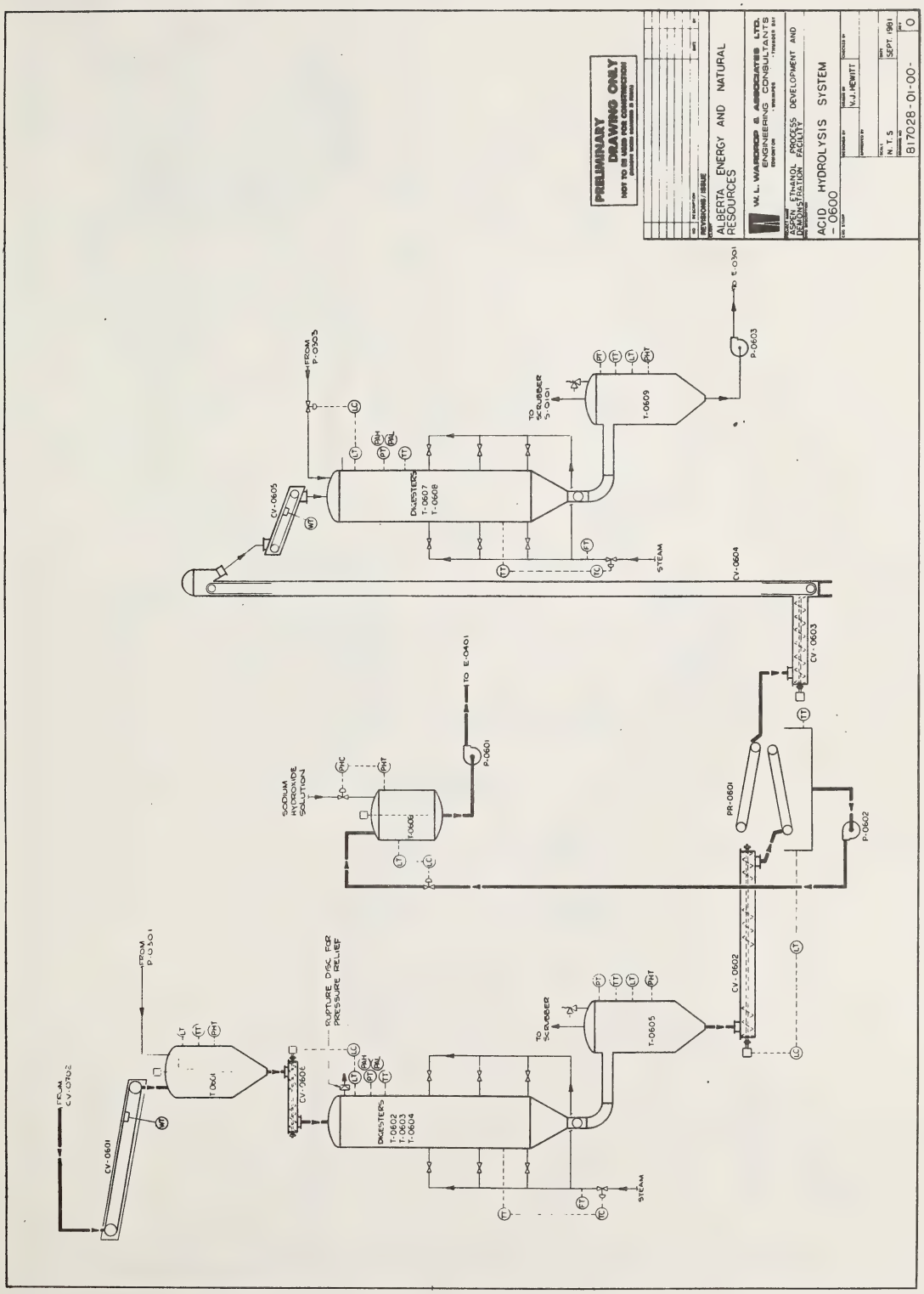

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ACID HYDROLYSIS SYSTEM
0600
817028-01-00-
SEPT 1991
W.J. HEWITT
FROM CV-0702
CV-0601
FROM P-0301
T-0604
RUPTURE DISC FOR PRESSURE RELIEF
CV-0606
DIGESTERS T-0602 T-0603 T-0604
TO SCRUBBER
T-0605
STEAM
CV-0602
SODIUM HYDROXIDE SOLUTION
T-0606
TO E-0401
P-0601
PR-0601
P-0602
CV-0603
CV-0604
FROM P-0303
CV-0605
DIGESTERS T-0607 T-0608
TO SCRUBBER S-0301
T-0609
TO E-0301
P-0603



**FIGURE 11**

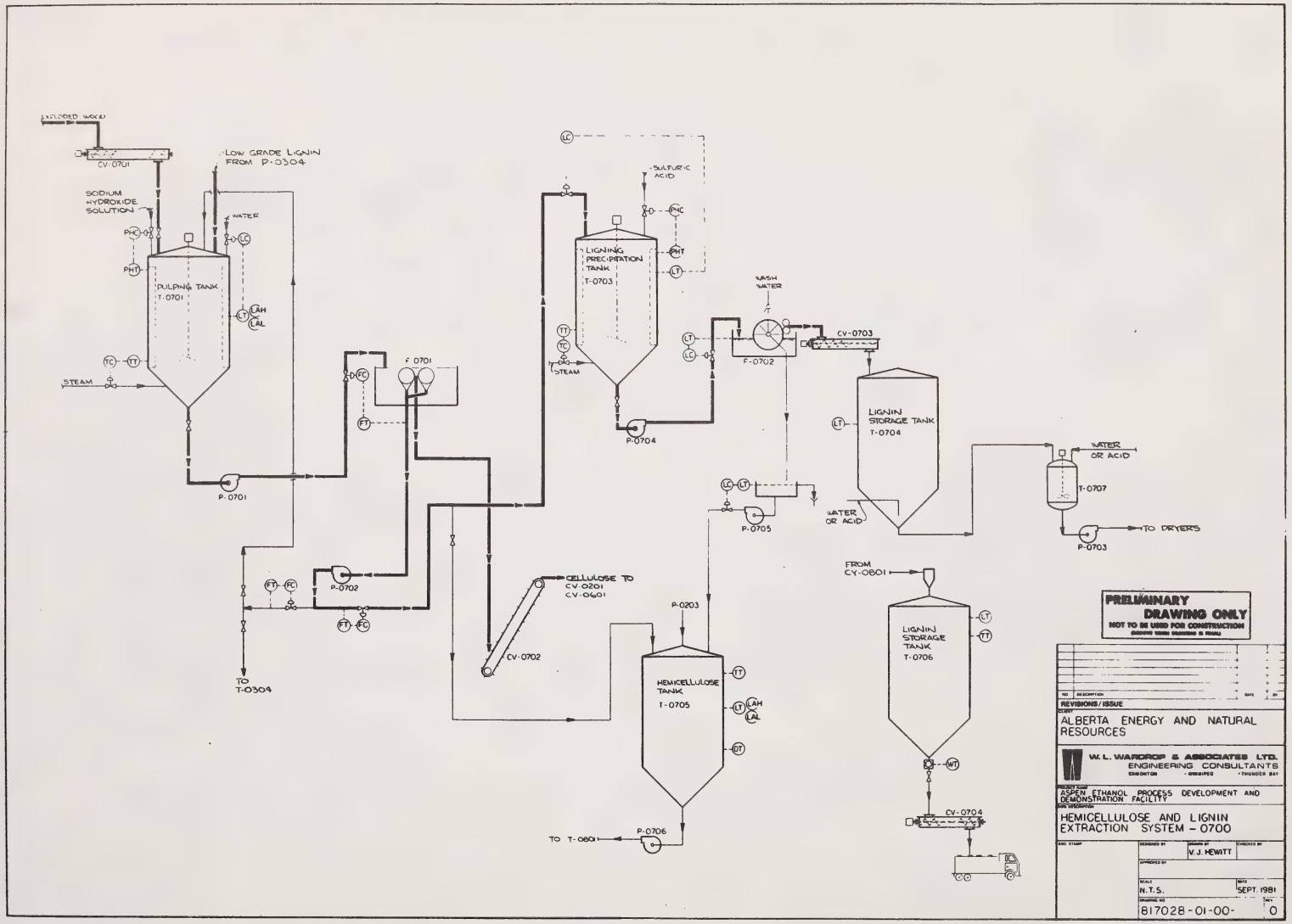

EXPLODED WOOD
CV-0701
LOW GRADE LIGNIN FROM P-0304
SODIUM HYDROXIDE SOLUTION
WATER
PULPING TANK T-0701
STEAM
P-0701
F 0701
P-0702
TO T-0304
CELLULOSE TO CV-0201 CV-0601
CV-0702
SULFURIC ACID
LIGNIN PRECIPITATION TANK T-0703
P-0704
WASH WATER
F-0702
CV-0703
LIGNIN STORAGE TANK T-0704
P-0705
WATER OR ACID
T-0707
TO DRYERS
P-0703
FROM CY-0801
LIGNIN STORAGE TANK T-0706
CV-0704
P-0203
HEMICELLULOSE TANK T-0705
P-0706
TO T-0801
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ASPEN ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY
HEMICELLULOSE AND LIGNIN EXTRACTION SYSTEM – 0700
DESIGNED BY
DRAWN BY V.J. HEWITT
CHECKED BY
APPROVED BY
SCALE N.T.S.
DATE SEPT. 1981
DRAWING NO 817028-01-00-
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FIGURE 12

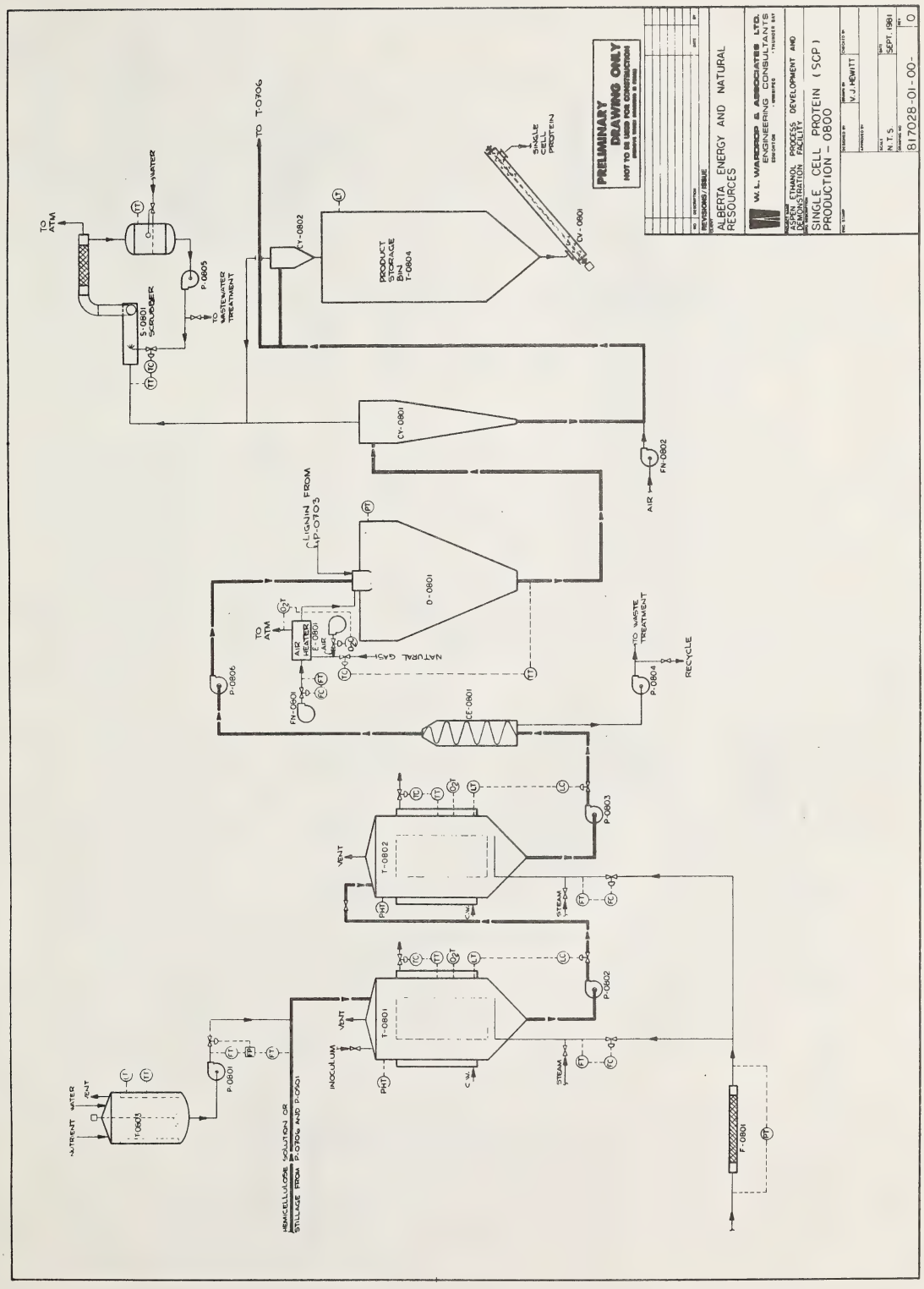

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ARGEN ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY
SINGLE CELL PROTEIN PRODUCTION – 0800
V.J. HEWITT
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TO T-0706
PRODUCT STORAGE BIN T-0804
SINGLE CELL PROTEIN
CY-0802
CY-0801
TO ATM
WATER
P-0805
TO WASTEWATER TREATMENT
S-0801 SCRUBBER
LIGNIN FROM LP-0703
D-0801
AIR HEATER E-0800
NATURAL GAS
FN-0801
FN-0802
AIR
TO WASTE TREATMENT
P-0804
RECYCLE
HE-0801
P-0806
T-0802
T-0801
P-0803
P-0802
VENT
STEAM
C.W.
INOCULUM
HEMICELLULOSE SOLUTION OR STILLAGE FROM P-0706 AND P-0701
TREAT WATER
NUTRIENT
T-0803
P-0801
P-0807



FIGURE 13

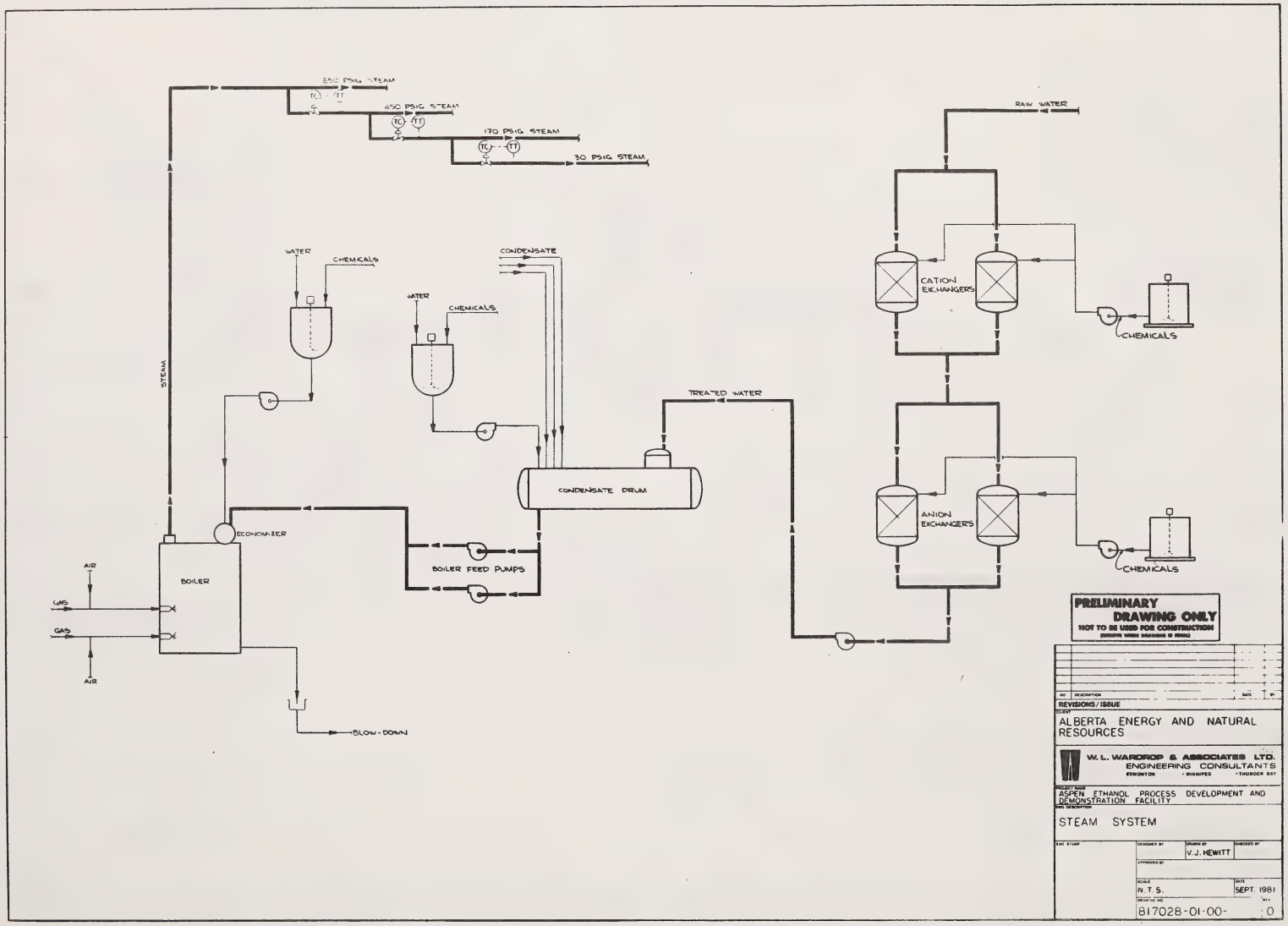

650 PSIG STEAM
450 PSIG STEAM
170 PSIG STEAM
30 PSIG STEAM
TC
TT
RAW WATER
WATER
CHEMICALS
CONDENSATE
CATION EXCHANGERS
CHEMICALS
STEAM
TREATED WATER
CONDENSATE DRUM
ECONOMIZER
BOILER FEED PUMPS
ANION EXCHANGERS
CHEMICALS
AIR
GAS
BOILER
GAS
AIR
BLOW-DOWN
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NO
DESCRIPTION
DATE
BY
CLIENT
ALBERTA ENERGY AND NATURAL RESOURCES
W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS
EDMONTON
WINNIPEG
THUNDER BAY
PROJECT NAME
ASPEN ETHANOL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY
DRG. DESCRIPTION
STEAM SYSTEM
ENG. STAMP
DESIGNED BY
DRAWN BY
V.J. HEWITT
CHECKED BY
APPROVED BY
SCALE
N. T. S.
DATE
SEPT. 1981
DRAWING NO
817028-01-00-
REV.
0

FIGURE 14

The Masonite gun pretreatment process is a batch operation. It takes approximately two minutes per cycle. Chips from the feed tank are charged into the Masonite gun and initially pretreated with low pressure steam to a temperature above the lignin's softening point. This operation takes 30 to 60 seconds. High pressure steam (approximately 4.1 MPa, 245°C) is then injected. Once the pressure in the reactor approaches that of the inlet steam, chips are released into a cyclone. This explosive decompression process disintegrates the lignocellulosic cells and converts chips into pulp. During this process, by-products, such as furfural, acetic acid and other volatiles are formed and are separated from the chips in the cyclone.

The Stake reactor achieves the same end result as the Masonite gun. However, it is operated continuously. Wood chips from the feed tank are fed to the reactor through a proprietary feeder. The feeder forms an airtight plug, permitting continuous feeding under pressure. The wood chips are treated at 200°C, for 6 minutes and these conditions are maintained by carefully controlled steam injection. The wood chips are discharged into the attached cyclone through a discharge screw and an orifice.

The Wenger extruder is different from the Stake reactor and the Masonite gun in that it uses only electricity as the process energy source, and high pressure steam is not required. Wood chips are fed into the reactor continuously. High temperature and high pressure are generated by the friction between the walls and the extruder screw. The extruder temperature approaches 246°C to 257°C with pressure ranging from 10 to 14 MPa. At the end of the pretreatment cycle, the wood chips are discharged into a cyclone through a restricted orifice. Volatile organic by-products such as furfural, generated during the pretreatment cycle, are separated in the cyclone and the treated wood recovered and stored in the surge tank for feeding to the hydrolysis section.

Volatiles, generated during explosive decomposition of wood, are trapped in a scrubber. Depending on their composition, these can be distilled to recover valuables such as furfural. No separation system is provided, as processes for optimum recovery of valuables should be developed after research and pilot-scale work.

## Enzyme Production and Hydrolysis

In this batch-operated system, enzymes are produced using the exploded wood substrate. The enzyme broth is then mixed with the substrate to achieve saccharification. Ten percent of the treated wood is first washed with water to remove the toxic materials and fed to an enzyme production tank. Trichoderma reesei is used for enzyme production and a two-step process is used for inoculum preparation. The culture (Trichoderma reesei) from the laboratory is first cultivated in the seed tank and then grown in the inoculum tank. Each step takes approximately 24 hours at 28 to 30°C. Culture from the inoculum tank is used to seed the enzyme production tank. At any given time, four tanks are used for enzyme production; one for hydrolysis and one is being cleaned. The enzyme production cycle requires 3 days at 28 to 30°C and a pH of 4. At the end of the cycle, treated wood is fed into the tank continuously for saccharification, which takes approximately 2 days (pH of 5, 45 to 50°C). After hydrolysis, the saccharified solution is held in a storage tank before feeding to the lignin separation section. A centrifuge is provided to recover the mycelia from the saccharified solution, which is recycled as nutrient for inoculum preparation.

Most of the equipment selected for this operation is conventional. A screw press is proposed for dewatering wash exploded wood, used for enzyme production, to obtain high washing efficiency.

## Lignin Separation

Lignin is recovered from the hydrolyzate by two-stage centrifugation with intermediate washing. The clarified sugar solution and the lignin wash are used for ethanol fermentation.

For acid hydrolysis, the lignin separation system (System 0300) forms an integral part of the hydrolsis process (System 0600). Thus, pulp from the second hydrolyzer is pumped to the first centrifuge, and solution recycled to the first hydrolyzer. Sulfuric acid solution and water are used for repulping the lignin and the centrate from the second stage centrifugation is recycled to the second hydrolyzer. Lignin recovered from this second stage centrifugation is stored in the low grade lignin storage tank. Further purification of the lignin is handled with the hemicellulose and lignin extraction systems (System 0700).

Centrifuges are selected for lignin separation as these have been used for lignosulphonate recovery at sulphite liquor ethanol plants. However, tests are required to determine their performance with native, or unreacted, lignin.

Fermentation System

A six-stage continuous fermentation system with yeast recycling is proposed for the conversion of sugar to ethanol. Sterilized air is injected into the first fermenter to revive the recycled yeast, and to generate fresh yeast. Excess air and carbon dioxide generated during fermentation are recompressed and sparged through the next fermenter for mixing. All the fermenters are jacketed to maintain constant (approximately 30°C) temperature. The pH in the fermenters is monitored and controlled at approximately 5. At the end of the fermentation, the beer passes through a hydrocyclone to remove the coarse particles before it is pumped to the yeast separator. The proper quantity of the recovered yeast is recycled to the first fermenter for maintaining high concentrations of acclimatized yeast. A fraction of the yeast is constantly purged along with the beer to prevent dead yeast build-up in the fermentation system. Beer from the yeast separator is stored in the beer well.

Distillation System

A three column conventional distillation process, offered by Vogelbusch Inc., is proposed as the reference process for ethanol recovery. Other azeotropic distillation systems are also available and can be considered during the process design stage. The Vogelbusch system contains a mash column, a rectifying column and a dehydration agent. Gasoline or cyclohexane are used as dehydration agent. Beer from the beer well (storage tank) is heated up in the dephlegmator using alcohol vapours and in a heat exchanger using stillage before it is fed into the mash column. Carbon dioxide in the beer is disengaged at the top of the mash column and the ethanol vapor fed into the rectifying column. The 96.4% volume ethanol (ethanol-water azeotrope) are fed into the dehydration column for 100% ethanol production.

Acid Hydrolysis

The acid hydrolysis process involves a two-stage counter-flow reaction between exploded wood and sulphuric acid. The first stage hydrolysis is performed at $150^{o}$ - $190^{o}$ C for 2-10 minutes and the second stage hydrolysis at $190^{o}$ - $250^{o}$ C for only 1-10 minutes. A batch operation is proposed to obtain high sugar concentrations. Three digesters are provided for the first stage and two digesters for the second stage. The treated wood from the pretreatment process is digested in the first stage with the acidic sugar solution recovered from the second stage digester. At the end of the first hydrolysis step, the cellulose sugar mixture is flashed and pressed using a belt press to recover sugar solution, which is neutralized for ethanol fermentation. Cellulose recovered from the press is charged to the next digester for second stage hydrolysis. Acid solution from the second stage centrifugation of the lignin separation section (0300) is used for hydrolysis. Hydrolyzate from the second stage hydrolysis is pumped to the lignin separation system (0300) for centrifugation, and recycled to the first stage digester.

Tests with the open-throat Moyno pump have shown that up to 30% solids slurry can be pumped using this equipment. Thus, continuous digestors could be used with substantial savings in capital and operating costs. However, detailed testing is required during process design to confirm the performance of Moyno pumps.

## Hemicellulose and Lignin Extraction Systems

Hemicellulose and lignin are extracted from the treated wood using sodium hydroxide solution in a continuous extraction system. This operation involves mixing the treated wood with a 2 percent sodium hydroxide solution at 70°C for 30 minutes in the extraction tank. The lignin bearing sodium hydroxide solution and the cellulose mixture from the tank are drawn continuously and pumped to a vari-nip press. The press recovers the cellulose and recycles it to the hydrolysis system for sugar production. The lignin-hemicellulose solution from the press is pumped to the lignin precipitation tank where sulfuric acid is added to obtain a pH of 4 at 80°C. Precipitated lignin and hemicellulose mixture is pumped to a rotary vacuum filter for lignin recovery. The lignin cake from the filter is stored in a surge tank and is dried using a spray dryer. The hemicellulose solution is used for single cell protein production.

## Single Cell Protein (SCP) Production

A two-stage continuous system is used to ferment the pentose sugar to produce single cell protein. An airlift fermenter with a draft tube in the centre is proposed for this operation. This fermenter has higher oxygen transfer characteristics and is particularly suitable for aerobic culture cultivation. The fermentation is operated at a pH of 5, and 35°C. Sterilized air is supplied to the fermenter through a sparger. Nutrients are added to the sugar solution before it enters the fermenter. Residence time in each fermenter is six hours. The fermented broth is centrifuged to recover the yeast (SCP) and the waste liquor from the centrifuge is treated in the plant's waste treatment system. The yeast (SCP) is dried to 90% solid using a spray dryer. A simple spray dryer is provided for both lignin and SCP drying.

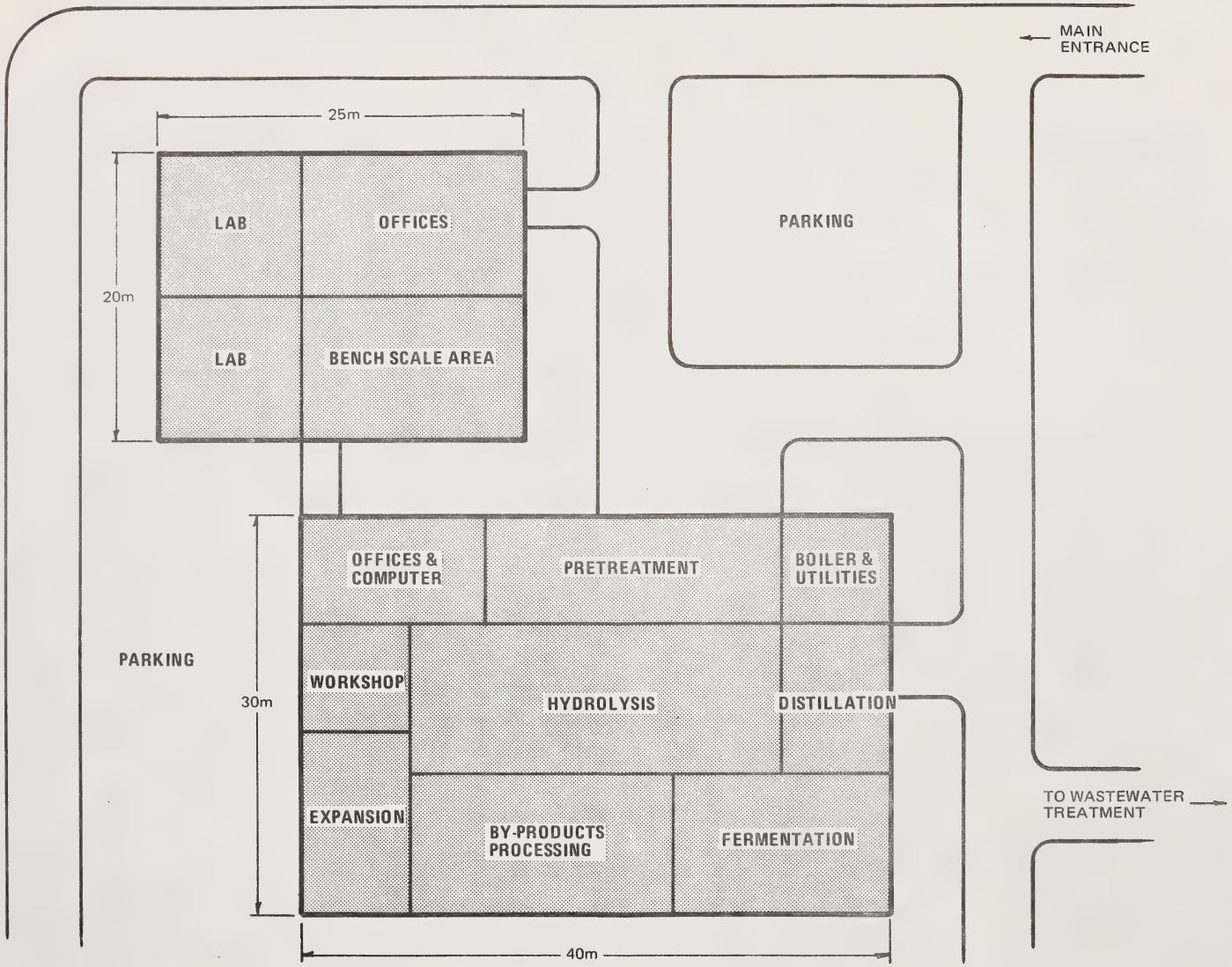



FIGURE 15

BUILDING LAYOUT

## Plant Description

### Plant Building

A proposed building layout is shown in Figure 15. The main building occupies 1200 $m^2$, and houses all process systems, utilities, control systems, workshop and offices for the operating staff. It is a steel clad, insulated building, 40 m long and 30 m wide. Heights are generally 15 m except distillation building, which is 25 m high.

The major process areas are isolated to facilitate environmental control, such as aseptic condition in hydrolysis and fermentation areas. Boiler room is located adjacent to distillation and pretreatment systems where most of the steam is used. Access is provided to workshop and process areas where product are stored or equipment and material are handled. Space is available adjacent to hydrolysis and fermentation areas for expansion.

Support laboratory facilities and administration offices are housed in a 500 $m^2$ building located adjacent to the main plant building. The laboratory will have the following components:

- Chemical
- Microbiological
- Bench-scale Area

The basic research will be performed at the laboratory, and its primary functions are:

- Testing cultures.
- Analyzing sample for chemical composition and chemical and physical properties.
- Assisting in process optimization.

In addition, the laboratory personnel will perform or assist with bench scale process development work.

## Control Philosophy

The basic objective of the proposed facility is to develop, demonstrate and provide scale-up data for the various technologies. In view of this function, high degree of process measurement, control and data logging capabilities are required. Thus all important process parameters will be monitored, and recorded in a central computer. Automatic controls will be provided for maximum number of process variables, and will be based on distributed control concept for flexibility and ease of addition of new process systems.

## Manpower

The proposed facility will employ a total of 42 persons. A breakdown is presented in Table 3, and the personnel fall under three categories:

1. Administration
2. Operations and maintenance
3. Technical Services

Operating personnel requirements are based on 5-days per week, 3 shifts per day operation. A total of 19 personnel are required for operation and maintenance, of which 12 are operators. Operations responsibilities are shared as follows:

1. Four first class stationary engineers for boiler, distillation and pretreatment systems.

2. Four operators for hydrolysis/fermentation.

3. Four operators for lignin and hemicellulose extraction and byproduct section.

TABLE 4

MANPOWER REQUIREMENTS

| | | |
|---|---|---|
| **Administration** | | |
| Plant Manager | 1 | |
| Administrative Officer | 1 | |
| Accountant | 1 | |
| Clerical Staff | 4 | |
| Security | 4 | |
| Sub-Total | | 11 |
| **Operation and Maintenance** | | |
| Operations Superintendent | 1 | |
| First Class Stationary Engineers | 4 | |
| Fermentation and Hydrolysis Operators | 4 | |
| Byproduct Systems Operators | 4 | |
| Maintenance Personnel | 2 | |
| Unskilled Personnel | 4 | |
| Sub-Total | | 19 |
| **Technical Services** | | |
| Technical Services Director | 1 | |
| Chemical & Biochemical Engineers | 2 | |
| Chemists & Bilogists | 2 | |
| Mechanical/Electrical Engineer | 1 | |
| Instrumentation Technician | 1 | |
| Analysts | 4 | |
| Non-skilled Workers | 1 | |
| Sub-Total | | 12 |
| TOTAL | | 42 |

The technical services department will include laboratory services and specialists in various processes and systems. These personnel will assist in formulating plant operation program, in trouble-shooting, and in planning modifications and additions to the facility as the form of ethanol production technology changes.

Conclusions

1. A 10 t/d capacity plant is required to achieve the objectives of the facility.

2. The plant building will cover 1200 $m^2$, and administration and laboratories an additional 500 $m^2$.

3. Basic function of the laboratory will be quality control and assistance in process optimization.

4. The plant will employ 42 full-time personnel.

COSTS

## Capital Cost

The capital cost for a demonstration plant with 10 t/d of BDWE capacity is estimated at $29.5 million. Capital costs for a 25 t/d BDW plant have also been estimated, and amount to $40.4 million. A breakdown is summarized in Table 5, and detailed capital cost estimates are presented in Appendix G.

The following basis have been used for capital cost estimation:

- All costs are expressed in 1981 Canadian dollars.

- The process system costs are estimated following Gutherie's "Chemical Process Modules" method.(2)

- Most equipment costs in the process systems are obtained from the supplier. Exceptions are Stake's reactor and the Masonite gun for which quotations were not provided. Costs of these equipment were then estimated using standard methods.

- Equipment installation and indirect cost are based on Wardrop's established factors for ethanol plants and Reference 2.

- Building and site services costs are based on unit factors developed for construction in Alberta. These include all building services and finishes.

- All laboratory equipment costs are based on catalogue prices.

- Utilities include boiler, steam distribution, cooling water, compressed air and off-site electrical. These have been estimated using methods similar to the process system.

TABLE 5

DEMONSTRATION PLANT CAPITAL COSTS

| | 10 t/d BDWE | 25 t/d BDWE (K$) |
|---|---|---|
| **Direct Costs** | | |
| Site Work Land | $ 300,000 | $ 400,000 |
| Serviced Buildings | 3,000,000 | 3,500,000 |
| Process Systems | 11,112,000 | 17,240,000 |
| Laboratory Equipment | 1,230,000 | 1,230,000 |
| Utilities (including off-site electrical) | 1,903,000 | 2,500,000 |
| Controls & Computer | 1,010,000 | 1,010,000 |
| Workshop and Office Equipment | 500,000 | 500,000 |
| Wastewater Treatment | 200,000 | 400,000 |
| Sub-Total | $19,255,000 | $26,780,000 |
| Contingency (20% TDC) | 4,845,000 | 6,720,000 |
| Total Direct Cost (TDC) | $24,100,000 | $33,500,000 |
| **Indirect Costs** | | |
| Engineering | $ 2,400,000 | $ 3,300,000 |
| Construction Management | 1,200,000 | 1,600,000 |
| Commissioning, including Computer Software | 1,300,000 | 1,500,000 |
| Studies and Approvals | 500,000 | 500,000 |
| Total Indirect Costs | $ 5,400,000 | $ 6,900,000 |
| TOTAL CONSTRUCTION COST | $29,500,000 | $40,400,000 |

NOTE: All costs in 1981 $ Canadian.

- The control and computer cost are in excess of those included as part of the process module. These are based on supplier quotations and installation costs based on Wardrop's experience.

- No allowances have been provided for interest during construction and working capital.

- Commissioning allowance covers the initial effort required in starting up a demonstration facility. Bulk of the commissioning costs, not included in this allowance, will have to be budgeted as operating costs during the later stages.

Operating Costs

Estimated annual operating cost for a 10 t/d BDWE plant is $2,500,000 in 1981 Canadian dollars and $3,520,000 for a 25 t/d BDWE plant. Details are presented in Table 6. Following basis are used for estimation:

- The plant will be operated 200 days/year, 5 days/week, 3 shifts per day.

- Raw material costs are based on $40 t/d BDWE, F.O.B. plant.

- 1981 Edmonton rates have been used for estimating water, electricity and natural gas costs.

- Wages for the plant personnel include a 20 percent overhead allowance.

- The maintenance cost is estimated at an average of 2 percent of the total construction cost. These costs are expected to be a substantially higher proportion of installed cost for equipment, and a low fraction for buildings and services.

- Miscellaneous costs include allowances for insurance, site maintenance, local taxes, etc.

TABLE 6

OPERATING COSTS

| | 10 t/d | 25 t/d |
|---|---|---|
| Raw Materials | $ 80,000 | $ 200,000 |
| Chemicals | 239,000 | 598,000 |
| Water | 6,000 | 14,000 |
| Electricity | 130,000 | 200,000 |
| Natural Gas | 30,000 | 75,000 |
| Wages | 1,315,000 | 1,383,000 |
| Maintenance | 600,000 | 900,000 |
| Miscellaneous | 100,000 | 150,000 |
| TOTAL | $2,500,000 | $3,520,000 |

NOTE: All costs in 1981 $ Canadian.

Other Costs

The capital and operating costs presented earlier are for the demonstration facility only. Additional costs will be incurred for the following:

- Management of the R & D program
- R & D funding.

The first item will depend on the management structure selected and should be determined as part of a management study. R & D funding is expected to start at a low value, and should grow with the program. A first task of a management group should be to define an R & D program, set priorities and define costs. At this time, we would suggest $500,000 per year in 1981 dollars for budgeting purposes.

Sensitivity of Cost to Size

Capital and operating costs have been estimated for the following two plant capacities to determine the variation of plant cost with size.

1. 10 t/d BDWE (recommended)
2. 25 t/d BDWE (alternate)

It should be added that the pretreatment system capacity for the 10 t/d plant are 25 t/d because of the availability of suitable equipment and desired reliability of scale-up data.

The costs are summarized below:

| | Capital Cost ($ million) | Operating Cost ($ million) |
|---|---|---|
| 10 t/d | 29.5 | 2.50 |
| 25 t/d | 40.4 | 3.52 |
| Ratios | 1.37 | 1.41 |

For a 250 percent capacity increase, cost increases are only 140 percent. This is below the average scale-up factor experienced in the chemical industry, which would range from 1.5 to 2.0 depending on the type of process.

The factors established in the industry are however applicable to large scale commercial facilities. For a small plant, costs of most major equipment remain practically unchanged, as labour cost savings become negligible. Savings in materials costs depend on the type and quality of materials and construction methods, and our discussions with equipment manufacturers have lead us to believe that even these savings are insignificant. Costs of a number of systems, such as instrumentation and control, remain unchanged or are only slightly affected. Another example is piping, for which materials costs are only a small fraction of total costs at low capacities.

Similar to the capital costs, increase in operating costs is relatively small as manpower requirements remain unchanged.

Revenues

The proposed 10 tonne per day BDWE plant will produce:

| | | |
|---|---|---|
| Ethanol | = | 475 tonnes/year |
| Lignin | = | 370 tonnes/year |
| Food Yeast | = | 75 tonnes/year |

Depending on the quality and market for these products, plant revenues could approach $400,000 per year. This would reduce the annual operating cost to 2.1 million in 1981 dollars.

Methods of Capital Cost Reduction

In the event that it becomes desirable to construct the facility for less than the conceptual estimate, we wish to outline the methods of reducing the capital costs:

| | | |
|---|---|---|
| 1. | In place of the three pretreatment systems proposed, provide one pretreatment system with room for installing an additional system on a temporary basis for testing. We do not foresee any major negative consequences from this action. Potential savings: | $ 2,600,000 |
| 2. | Also, simplify lignin separation system. This could result in a higher sugar loss with lignin and lower sugar concentration. Potential savings: | $ 500,000 |
| 3. | Also, simplify lignin and hemicellulose extraction system. Consequences could be lower yield of high quality native lignin and low sugar concentration. Potential savings: | $ 800,000 |
| 4. | Eliminate food yeast production system and use ethanol fermentation system to produce food yeast. This could limit flexibility of continuous process demonstration and production of fermentation chemicals other than ethanol. Potential savings: | $ 1,200,000 |
| 5. | Eliminate computer and reduce level of sophistication in instrumentation and control. | $ 1,000,000 |
| | Total Potential Saving (1981 $) | $ 6,100,000 |
| | Revised 1981 Cost Estimate | $23,400,000 |

We do not believe that the above cost reductions seriously compromise the objectives of the facility. A reassessment of program objectives and plant concept is desirable if further reductions are necessary.

## Proposed Plant Budget

The capital costs discussed in the previous sections were in 1981 dollars. However, actual plant construction is expected to take place during 1982-83 period. To ensure adequate provision from a budgeting standpoint, we recommend that an annual escalation of 14 percent be included in the cost. This will result in a budget figure of $37 million for the proposed facility.

Recommended budget of facility with a reduced scope and flexibility is $30 million.

## Conclusions

1. Capital and operating costs for a 10 t/d facility are estimated at $29.5 million and $2.5 million per year respectively in 1981 dollars. Expected annual plant revenue is $400,000, reducing the annual operating cost to $2.1 million per year.

2. Costs for overall management of the program and the R & D should be determined during the proposed management study. We recommend a budget allowance of $500,000 in 1981 dollars.

3. Capital cost increases with size are lower than the industry average because of the small plant capacity.

4. A facility with a somewhat reduced scope and flexibility can be built for $23.5 million in 1981 funds.

5. Recommended capital budget allowance for construction in 1982-83 is $37 million. Budget allowance for a facility with a reduced scope is recommended at $30 million.

# POPLAR CHEMICAL PROCESS DEVELOPMENT AND DEMONSTRATION FACILITY - SUPPLEMENT ON ORGANOSOLV SACCHARIFICATION


## SUMMARY

Organosolv saccharification process for the conversion of wood to sugars has a number of attractive features such as high sugar yields and recovery of high quality lignin. This supplement to our report on the Poplar-chemical Facility provides a conceptual design and cost of a 10 t/d BDWE system based on the process developed by Dr. Laszlo Paszner at the University of British Columbia.

Based on the laboratory test data, sugar yields in the range of 85 to 90 percent and lignin recoveries approaching 100 percent have been assumed. Major system components are: a high pressure chip feeder; continuous counter-current digester and solvent recovery system. Sugar solution containing lignin will be treated in the process units already discussed in our report. No major change in the existing conceptual plant layouts, manpower and services is required as a result of this addition; the only exception being an increase in the boiler and cooling tower capacities because of the higher energy consumption over other pretreatment processes.

We estimate the capital cost of the demonstration facility incorporating organosolv process to be $31.4 million in 1981 dollars. Suggested budget allocation is $39 million. Estimated annual operating cost is $2.57 million in 1981 dollars.

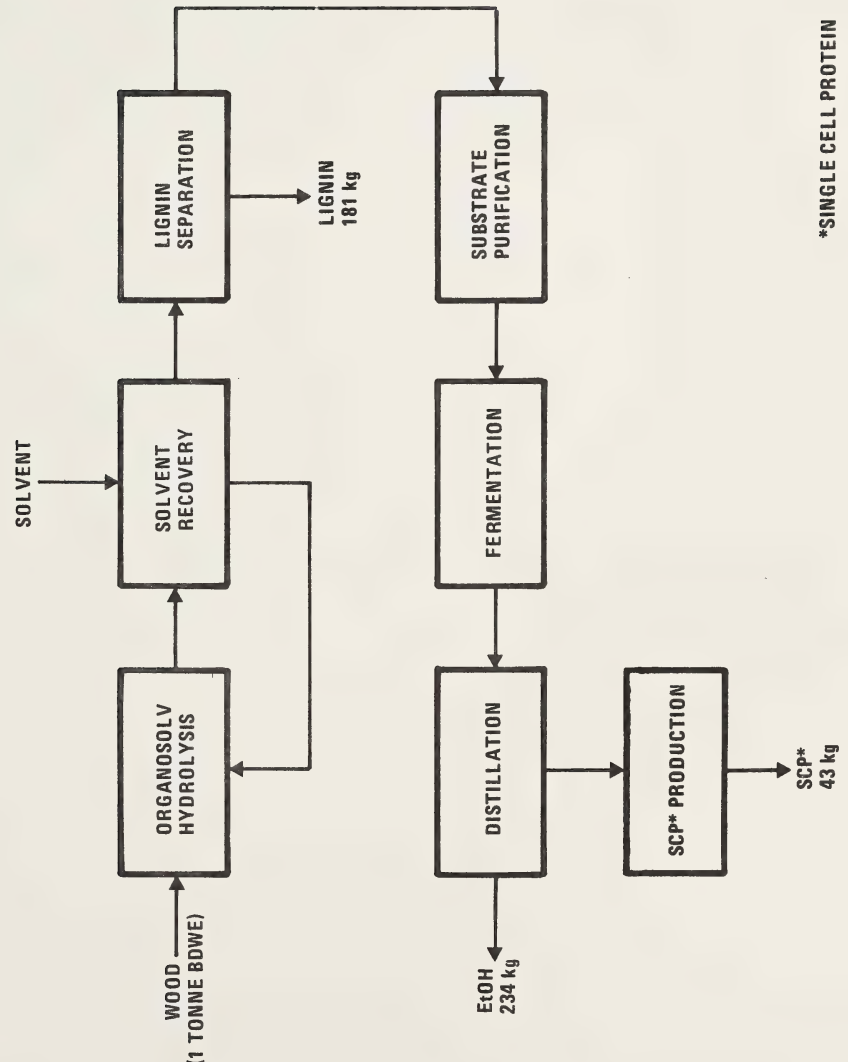
The next stage in the development of the organosolv process proposed by Dr. Laszlo Paszner is bench-scale testing in a semi-continuous unit. This should provide adequate data for the design of a 10 t/d BDWE demonstration unit.

## INTRODUCTION

The Alberta Energy and Natural Resources is planning to build a demonstration facility for making chemicals from poplar through pretreatments, hydrolysis and fermentation. A report was submitted by W.L. Wardrop & Associates Ltd. in March, 1982, on the conceptual design of the facility. It covered a review of the state-of-the-art, the selection of the plant processes, conceptual design of process system and plant, estimation of the cost of construction and operation, some concepts of how the facility may be managed, review of markets for the products and the development of a research and development program.

Three pretreatment systems, which have been either commercialized or demonstrated on an industrial scale, were included in our report. A new wood to sugar production route - organosolv process - developed by Dr. Laszlo Paszner at the University of British Columbia has generated high level of interest because of its ability to achieve high sugar yields and recover high quality lignin. AENR has asked Wardrop to develop the cost of a 10 t/d BDW organosolv saccharification unit for installation at the demonstration plant. This supplement to our report describes the process, costs and R&D needs for the organosolv system.

The laboratory data provided by Dr. Laszlo Paszner has been used for developing the process flow diagram and equipment selection, and we expect the accuracy of our cost estimats to be within $\pm$35% instead of the $\pm$25% for the initial concept. Further bench-scale work is required for developing more accurate concept and costs.






ORGANOSOLV PROCESS - MATERIAL BALANCE

FIGURE 1

## INTEGRATION INTO THE PROPOSED PLANT BUILDING

The addition of the organosolv hydrolysis to the plant will not change the building layout proposed previously. It will be located in the hydrolysis section where adequate space exists.

No change in manpower requirements and services is expected, except an increase in the boiler and cooling tower capacities because of the high energy consumption by the organosolv saccharification process.

COSTS

The capital cost for a 10 t/d BDWE demonstration plant with additional organosolv hydrolysis process is estimated at $31.4 million in 1981 dollars compared with the $29.5 million for the concept presented in our report. A breakdown of the revised plant capital cost is presented in Table 1 and the detailed costs of the organosolv process are presented in the Appendix.

The revised plant budget is $39 million for a facility incorporating organosolv process.

Estimated annual operating cost for a 10 t/d BDWE with organosolv process is $2.57 million expressed in 1981 dollars. The details are presented in Table 2. Cost increases result mainly from an increase in energy consumption and the solvent used in the process.

TABLE 1

REVISED DEMONSTRATION PLANT CAPITAL COSTS

(ORGANOSOLV PROCESS)

| Direct Costs | 10 t/d BDWE |
|---|---|
| Site Work Land | $ 300,000 |
| Serviced Buildings | 3,000,000 |
| Process Systems | 12,348,000 |
| Laboratory Equipment | 1,230,000 |
| Utilities (including off-site electrical) | 1,923,000 |
| Controls & Computer | 1,010,000 |
| Workshop and Office Equipment | 500,000 |
| Wastewater Treatment | 220,000 |
| Sub-total | $20,531,000 |
| Contingency (20% TDC) | 5,169,000 |
| Total Direct Cost (TDC) | $25,700,000 |
| **Indirect Costs** | |
| Engineering | $ 2,600,000 |
| Construction Management | 1,300,000 |
| Commissioning, including Computer Software | 1,300,000 |
| Studies and Approvals | 500,000 |
| Total Indirect Costs | $ 5,700,000 |
| TOTAL CONSTRUCTION COST | $31,400,000 |

NOTE: All costs in 1981 $ Canadian.

TABLE 2

REVISED OPERATING COSTS (ORGANOSOLV PROCESS)

(1981 $)

| | 10 t/d |
|---|---|
| Raw Materials | $ 80,000 |
| Chemicals | 153,000 |
| Water | 6,000 |
| Electricity | 136,000 |
| Natural Gas | 75,000 |
| Wages | 1,315,000 |
| Maintenance | 600,000 |
| Miscellaneous | 100,000 |
| TOTAL | $2,565,000 |

RESEARCH AND DEVELOPMENT NEEDS

The Organosolv Process, proposed by Dr. Laszlo Paszner, has been developed and tested to date mainly in the laboratory. The next stage in its evolution is bench-scale testing using a semi-continuous digester. Parameters that need to be determined are:

- Optimum solvent to acid catalyst ratio
- Optimum temperatures and residence times
- Sugar yields, and lignin recovery efficiency
- Solvent degradation and losses.

Sufficient kinetic and design data should become available as a result of this work to design a continuous digester. Parallel work may be required, specially in the design of a chip feeder and kinetics of wood dissolution if continuous reactor is to become a reality. Further optimization of process conditions can then be performed on the pilot stage.

CONCLUSIONS

- Capital cost and operation cost for a 10 t/d BDWE facility incorporating the organosolv process proposed by Dr. Paszner are estimated at $31.4 million and $2.57 million per year respectively in 1981 dollars. Suggested capital budget allocation is $39 million.

- The additional organosolv process will not increase the manpower requirement, nor will it change the building layout.

- The next logical step in the development of the organosolv saccharification process is bench-scale testing in a semi-continuous reactor.

MANAGEMENT OF THE FACILITY

## Introduction

The primary objective of the facility is to promote the development and commercialization of technologies for making ethanol and other chemicals from poplar through pretreatment, hydrolysis and fermentation, with the ultimate goal of achieving better utilization of Alberta's poplar resource.

This objective should be achieved by:

1. Alberta taking a leadership role by formulating a technology development program, and by funding research and development efforts covering specific processes, as well as areas affecting the overall economics of ethanol production (e.g. by-product processing).

2. Making the demonstration plant facilities and manpower available, at mutually agreeable terms, to the private sector for demonstrating technologies and obtaining scale-up data.

A management structure is required to ensure that these objectives are achieved.

## Major Issues and Experiences of Other Organizations

Major issues involved in the operation of such a facility are expected to be:

1. Establishment of a policy on ownership, licensing and marketing of technology.

2. Creation of an environment which would encourage the private sector, especially process developers, to use the facility for its intended purpose.

3. Development of policies regarding funding of research on laboratory, bench and pilot plant scales.

The two-pronged approach of encouraging private industries to participate in the use of the demonstration plant while funding research on processes and owning technology, although not totally exclusive, may be difficult to reconcile. Experience has shown that a private process developer can be suspicious of a large research organization with significant funds. A number of organizations in Canada and the U.S. have faced this situation. Discussions were held with the following:

1. Alberta Oil Sands Technology and Research Authority (AOSTRA)
2. Alberta Research Council (ARC)
3. POS Limited, Saskatoon
4. British Columbia Research Council (BCRC)
5. National Research Council of Canada (NRC)
6. Solar Energy Research Institute, Golden, Colorado (SERI)

A brief discussion of the objectives of these organizations and their management structures follows:

Alberta Oil Sands Technology and Research Authority (AOSTRA)

This Provincial Crown corporation was formed in 1975 by an Act of the Government of Alberta. The basic objective is to promote the development of technologies for the utilization of oil sands, heavy oil and enhanced recovery of conventional crude oil. AOSTRA has an annual budget of approximately $40 Million. The corporation's activities can be broken down into two major areas:

1. Basic research
2. Applied research including pilot plant work

AOSTRA finances basic research in Canadian universities and research centres, and in such cases, ownership of technology rests completely with AOSTRA. This technology is then licensed to the private industry to generate funds for further growth. Normally know-how may be transferred immediately without complete development to speed up technology growth. Recently, licensing of such technology has started to generate revenues to cover administrative costs.

The corporation also funds pilot projects jointly with the private industry in recovery and processing. Although the ownership and licensing rights for such technologies remain with AOSTRA, prior technology is recognized and income-splitting formulas are developed based on prior technology and developers' contribution. Generally, recovery and upgrading technologies may not be licensed until a complete system is developed for marketing on a royalty basis. However, there have been some exceptions when partially developed technologies have been licensed.

Normally, applications for funding are reviewed by a peer group and recommended to the Board of Directors. The policies on proposal evaluation, funding and technology ownership were developed after input from industry and the research community. This has helped the corporation in achieving its objectives.

During a telephone conversation, Dr. Rick Luhning of AOSTRA, expressed the opinion that if significant sums of money are to be invested over a long period of time, an organization such as AOSTRA would be quite suitable for development of Poplar-Chemical technologies. In answer to a question regarding subsidiaries of an existing corporation, he felt these may be easier to set up for a low budget, short-term program.

Alberta Research Council (ARC)

Alberta Research Council, a Crown corporation founded in 1921, operates under the Research Council Act. This legislation gives the Council a mandate to advise the government on scientific matters and to promote responsible economic development through the use of science and technology.

In a meeting with Dr. G.G. Cloutier, the President of Alberta Research Council, he indicated that the ARC is interested in managing the research and development program. He stressed that it would be preferable to involve private industry, users of the technology, in the ownership, management and operation of the facility, as this will be consistent with the ARC charter.

Discussions were also held with other Alberta Research Council personnel regarding the Council's policy on technology ownership, its dealings with private industry, and its potential role in ethanol process development through the proposed facility. The following points were raised:

1. ARC is actively engaged in technology development and deals regularly with conflict of interest situations. It owns patents and licenses technology, and also performs contract work for the private sector under secrecy agreements.

   Technology developed under contract is owned by the industry. Potential conflict of interest questions have been avoided by negotiating contracts satisfactory to the industry, and following the letter and spirit of these contracts rigorously.

2. ARC prefers to work on specific projects rather than on long-range broad development programs. It would, however, be prepared to assume the responsibility for developing a specific process from laboratory to demonstration stage.

3. ARC does not have access to adequate laboratory space at this time. January 1984 is the expected date of completion of a new building, although some delays may occur.

POS Limited

This pilot plant facility was established with joint federal-provincial funding to conduct in-house research and to provide research and development service to the food industry. Private industries throughout the prairies have membership in the company. Any processes developed are first offered to the membership for six months. After six months, these can be marketed elsewhere by POS.

Any contract work done for the industry is covered by confidentiality agreements, which are vigorously followed. Only the Director of the Centre and the Project Manager know the complete detail of a project. In the past, confidentiality has been a concern for some of the companies, but all possible steps have been taken to establish their confidence. It is felt that rigid structures separating contract work from in-house R & D are not essential to protect clients' interests.

As the centre shares in-house developments with the entire membership, individual members with saleable ideas have not come forward. Steps are now being taken to remedy this situation.

British Columbia Research Council (BCRC)

The BCRC has a similar role in B.C. as the ARC in Alberta in the areas of technology development and ownership. Rigorous compliance with confidentiality contracts has avoided potential conflicts with clients.

Ten years ago, the Council formed a limited company - Techwest - wholly owned by the Council to commercialize proprietary technology. However, they have been unable to establish this firm as a profitable entity because of the difficulties in capitalization.

In future, the preferred route for commercialization of BCRC technology will be partnership with private enterprise, with the objective that the private sector will ultimately buy out BCRC.

National Research Council of Canada (NRC)

National Research Council has extensive in-house programs in research and development of a wide range of technologies. Financial assistance programs exist for transferring these technologies to the private sector for further development and commercialization. PILP (Pilot Industry Laboratory Program) is an example of such programs. In this case, NRC can fund development work by a private industry based on NRC expertise, under contribution agreements. Patents from such development work are completely owned by the firm. Recently, the program has been expanded to include technologies developed by other federal agencies.

NRC can also fund research under procurement contracts. However, all results and technology are owned by NRC in this case.

Solar Energy Research Institute, Golden, Colorado (SERI)

SERI is presently responsible for funding all research in alcohol fuels on behalf of the United States Department of Energy. The Institute also has its own R & D program in biotechnology and alcohol fuels. Despite such potential conflict of interest the Institute has been quite successful in attracting private industry. An example is Iotech's use of Gulf's pilot plant under a DOE contract.

SERI's success has been attributed to the following:

1. Enunciation of a clear policy regarding the ownership, licensing and commercialization of technology. DOE owns the patent, but the commercial developer has non-exclusive license. However, if commercialization is not achieved within a period considered adequate by DOE, licensing rights may be revoked.

2. By separating the contractual and project management functions from R & D functions to ensure protection of information by suitable organizational structure.

In SERI's opinion, clear-cut policies regarding secrecy and ownership must be established if participation by private industry is desirable.

## Discussions

The above investigations indicate that a number of organizations have succeeded in carrying out a dual role of in-house research, and contract research and funding of process development work through contractors quite successfully in the past. The proposed R & D strategy for Poplar-Chemical technology development would not involve in-house research at the pilot plant and the potential of conflict of interest should be less severe than that experienced by SERI or POS.

AOSTRA model has been very successful in achieving the goals of advancing technology in alternate petroleum fuels development. Similar structure should be considered for the R & D portion of the Poplar-Chemical technology development program.

## Management Alternatives

The following management alternatives are available.

1. Management by a Crown corporation.

2. Management by Alberta Research Council.

3. Management of the overall program by a working group, lead by AENR, implementing its policies through Alberta Research Council, with operation of the demonstration plant by private industry under contract to ARC.

4. Management by a form of consortium between government and industry.

ALBERTA ENERGY & NATURAL RESOURCES
POPLAR - CHEMICAL TECHNOLOGY AUTHORITY
PILOT PLANT OPERATIONS DIVISION
PROJECTS DIVISION
R & D DIVISION
1. Responsible for Plant Operation
2. Defines Plant Operations Objectives
3. Directs Process Developers to R & D Division if Process not ready for Demonstration
4. Assists Projects Department in negotiations with Process Owners wishing to use the pilot plant
1. Assists in formulating technology ownership policies
2. Negotiates with process owners and research contractors
3. Manages research & pilot scale projects
4. Could control flow of information between contractor and R & D group where confidentiality requirements arise
1. Responsible for basic Research & process development funded by Alberta
2. Defines R & D programs objectives
3. Formulates policies regarding technology ownership & marketing
4. Funds basic research to universities and research laboratories
5. Could delegate specific pilot & demonstration scale process development to Alberta Research Council

ALTERNATIVE 1
MANAGEMENT BY A CROWN CORPORATION



**FIGURE 16**

## Management by a Crown Corporation

The proposed structure for this alternative is shown in Figure 16. This option provides a single organization with adequate control over both operation and R & D functions. Potential questions regarding conflict of interest and unlawful dissemination of information are resolved by a clear separation between the two functions; by negotiating mutually agreeable contracts on specific projects; and by demonstrating to the private industry that these contracts are rigorously followed.

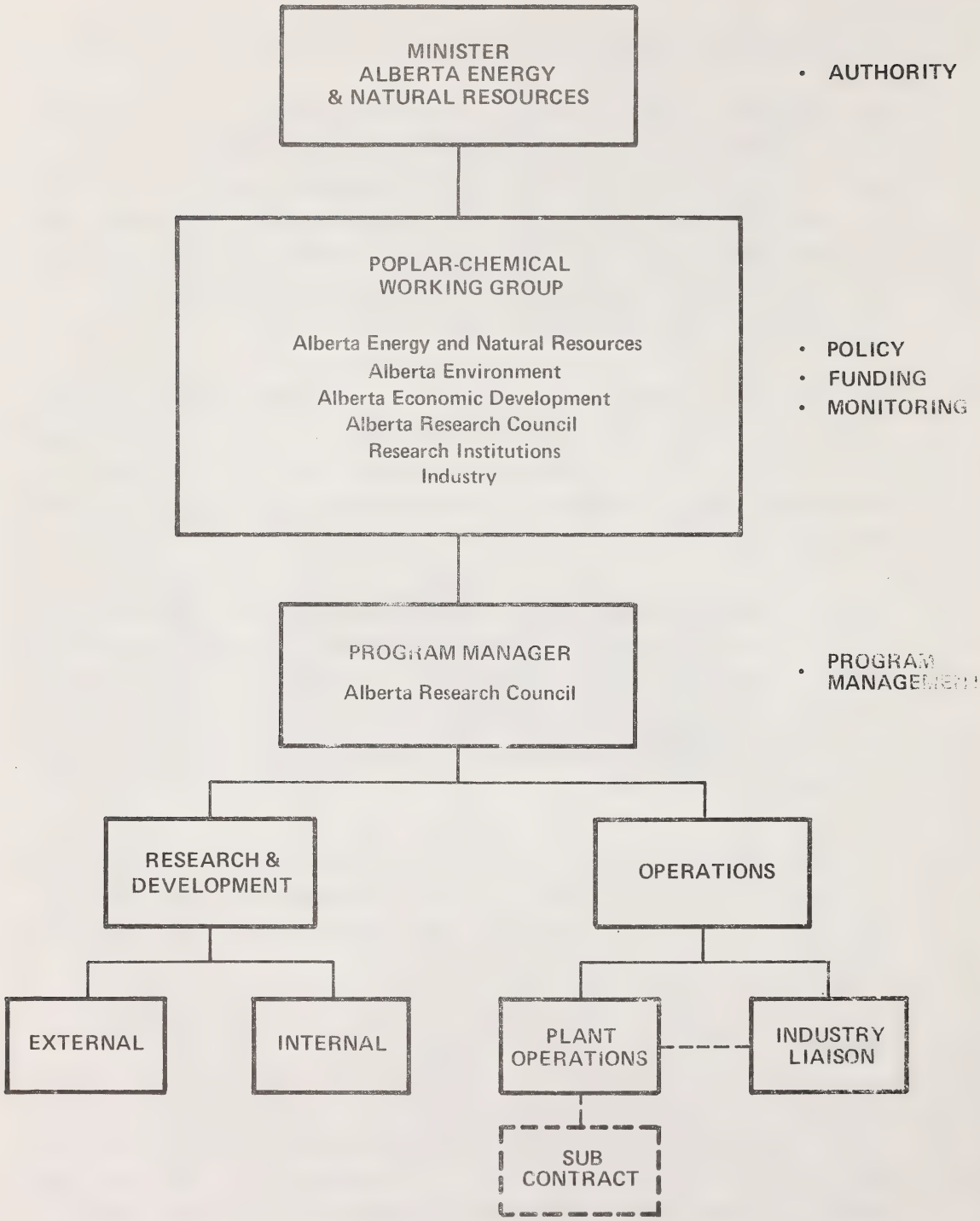
The department responsible for research and development could follow the AOSTRA model to fund basic and applied projects and to license technology. Input from the research community and private industry can be sought in formulating the R & D program to ensure applicability of the technologies developed.

It may be expedient to set up the proposed structure as a subsidiary of an existing Crown corporation. Examples of potential parent corporations are AOSTRA and ARC. The Federal government has adopted this route by setting up Canertech as a subsidiary of Petro-Canada. Setting up of a subsidiary of an existing Crown corporation would require a revision of the existing Acts governing these bodies.

However, the administrative cost of such an organization is expected to be in the order of $700,000 annually. It can be argued that these funds can be best spent on actual R & D rather than administration. This option is not preferred because of its high costs.

## Management by ARC

Our discussions with ARC indicate that they are willing to play an active role in this program. However, their preference will be to participate with industry, who will be the users of this technology. They could manage the R & D program, but would like the industry to be involved in plant operation management.




ALTERNATIVE 3 - MANAGEMENT BY A WORKING GROUP FIGURE 17

Management by a Working Group

An example of the management of the R & D program by a working group is the existing Forest Product Working Group through ARC. The program manager is from ARC. University and industry personnel are both represented in the group. Such a group could be well suited to directing and managing an R & D program to be contracted out to the universities and industry. Technology developed under such programs will be owned by ARC.

A potential management structure for this Alternative is present in Figure 17. Policy formulation and funding allocation functions are controlled by the Working Group. ARC manages the R & D and operations. However, plant operation could be contracted out to a private industry.

Major features of this approach are:

- it achieves separation of policy/funding functions from management;
- it is consistent with ARC mandate regarding industrial participation;
- it ensures government control with optimum private participation;
- it takes advantage of ARC experience in managing R & D programs;
- enabling legislation is already in place.

A potential problem may arise in the operation of the demonstration plant by a private firm. It may be difficult for such a firm to provide and retain experienced personnel because of the short-term nature of the contracts. Alternatively, ARC could provide the operating staff.

If properly defined and implemented, this approach can achieve the basic requirements for successful operation of the facility. The R & D program can be separated from the plant operation to satisfy process developers that their know-how will be protected.

## Management by a Government-Industry Consortium

The proposed pilot plant and the R & D programs could be managed by a consortium of government and private industry. Involvement by major forest resource industries, chemical industries and ethanol process firms could be sought. A number of models are possible for such an organization, but the following two structures are most relevant.

a) Organization similar to POS Limited in which private companies participate under a membership program.

b) Establishment of a non-profit organization under the Societies Act. Examples are Petroleum Recovery Institute and Computer Modelling Group. Government assistance is provided under Provincial or Federal/ Provincial programs to establish commercial viability of such ventures.

The direct involvement of industry in the R & D program is attractive from the standpoint of commercialization. The following points should be noted:

1. Ethanol or chemicals from wood may not be a high priority of the conventional forest product or chemical industry and the level of interest could be low. However, some industries, in very preliminary discussions, have shown an interest in the proposed facility and its operation and in-depth surveys are required to determine actual interest. Such surveys are beyond the scope of this study.

2. POS experience has shown that most of the major food industries are subsidiaries of U.S. firms with their own research activities at their head offices. Thus motivation for sharing research activities in Canada is not very high. Member companies should be carefully selected to avoid similar problems for the proposed program.

3. As this concept may require information disclosure to all members, conflict of interest questions may arise. This problem can, however, be minimized by adopting suitable policies.

4. A number of the firms involved in ethanol process development are small independents. They may be hesitant to cooperate with multinational corporations.

5. POS has experienced difficulties in raising operating funds, as it is not clear where the final responsibility lies.

Despite these potential problems, a consortium approach is expected to be the most suitable option, as industry involvement should speed up technology implementation. We believe that a suitable management structure can be developed which could maximize advantages of this approach. Such a structure should only be developed once the number, nature and interests of potential participants is known. A detailed management study is recommended to solicit input from private sector and to evaluate their level of interest in the proposed program.

## Conclusions

To achieve the program objectives, it is proposed that the operation function of the demonstration plant be clearly separated from the management of the basic research and development activities.

An industry-government consortium approach which would speed up commercialization of technology is desired to achieve program objectives. Detailed structure can best be defined after the number, type and interests of potential participants are known. A management study is recommended to determine the requirements and needs of such participation by the industry.

An alternative approach is the management of the R & D program by a working group composed of government, industry and university personnel, headed by AENR, and implementing its policies through ARC. The plant operation could be contracted out to the private industry.

MARKETS FOR DEMONSTRATION PLANT PRODUCTS

## Introduction

A preliminary survey has been performed to determine potential markets for the plant products. In addition, markets for secondary products, and lignin-based products with a high potential, have also been assessed. Details of our investigations are presented in Appendix H, and a brief discussion follows:

## Plant Products

Plant products include ethanol, lignin, fusel oil and single cell protein (yeast). Maximum quantity of each of these products for a 10 t/d capacity are shown below.

| | DEMONSTRATION PLANT (10t/d BDWE) | |
|---|---|---|
| | Acid Hydrolysis | Enzyme Hydrolysis |
| Ethanol, L/d | 3,000 | 3,250 |
| Lignin, t/d | 2.26 | 2.38 |
| Fusel oil, L/d | 13.4 | 14.5 |
| Single cell protein, kg/d | 290 | 375 |

Ethanol is the primary product of the demonstration plant. As an industrial solvent, it is used mostly in the printing trade and as a solvent or dilutant in compounded products such as cosmetics, perfumes, paints, inks, adhesives and automotive products.

The Canadian market for industrial ethanol is 79 million L/a. This includes all medical and pharmaceutical uses but also includes at least 6.8 million litres of lower grade ethanol than will be produced by the pilot plant. The maximum annual production from the demonstration plant of 650 000 L/a can probably be sold within the existing low grade industrial alcohol market in Canada as it represents less than 16 percent of this market.

The price for anhydrous denatured ethanol in tank car lots FOB Edmonton is estimated at $0.77 per litre, based on the drum price less a drumming charge. Thus the annual sales value of ethanol at maximum production rate is $500,000, in bulk, FOB Edmonton plant site.

Other potential markets for the fuel-grade ethanol produced at the plant are: as a gasoline extender and octane enhancer; and as an on-farm fuel. If these markets are developed, all plant production can be easily consumed. The most attractive fuel alcohol market in Canada is in Manitoba, where a provincial tax relief exists on gasohol, a mixture of 10 percent ethanol and 90 percent unleaded gasoline.

A number of markets exist for the modified lignin produced by the sulphite pulping industry. Native lignin produced by the plant can substitute some of the existing products. However, the long-term potential is in the conversion of native lignin into a host of secondary high value products. Their development requires further research and market tests. Initially, lignin can be burnt as a fuel in the plant boiler, and equipment should be provided for its handling and burning as a slurry.

The small quantity of single-cell protein should find a market as a human food supplement or as a cattle feed. Fusel oil can best be burnt in the plant boiler. Similarly, the plant boiler should be equipped to burn low quality lignin solutions, volatiles and other waste products.

Conclusions

1. A sound market exists for ethanol either as low-grade industrial alcohol in Alberta and elsewhere or as gasohol in Manitoba. The market is large enough to accept all of the demonstration plant production. Marketing is best done by long term contract.

2. The high quality lignin which will be produced at the demonstration plant is not presently available in commercial quantities. This will provide an excellent base material for production of vanillin, pharmaceuticals and other high value products. Marketing is best done by contract with major lignin marketers or lignin users. Research into the use of this form of lignin holds potential for an Alberta wood based chemical industry.

3. Single cell protein should be suitable for human consumption. Alternately, it will find a ready local market in animal feed formulations.

4. The remaining products should be used for product research but are in quantities too small to warrant the cost of marketing. The demonstration plant boilers should be equipped to burn fusel oil and possibly concentrated lignin solutions.

## SITE SELECTION CRITERIA

### General Criteria

The normal site selection criteria used for major industrial facilities include:

- Raw material availability
- Product markets
- Availability of labour
- Services
- Utilities and fuels
- Environmental considerations

### Raw Material Availability

The proposed demonstration plant will consume 10 t/d BDWE. This is approximately one truck load per day. It is expected that the wood will be harvested as part of an existing logging operation and distance of the demonstration plant from the raw material source is not an overriding factor in selecting a plant site.

### Product Market

The plant is estimated to produce an average of 475 t/a of anhydrous ethanol, 370 t/a of lignin and 75 t/a single cell protein. Because of the small quantities involved, proximity to the market is not considered to be very important.

## Availability of Labour

The plant will employ 40-50 full-time personnel. A significant number of these will be professionals and highly trained technical personnel and living conditions will be an important criteria. These include housing, schools, recreational facilities, health care, media representation, etc.

Plant location can significantly affect the availability and cost of construction labour. These factors must be considered during site selection.

## Services

These include services such as fire protection, police and security, maintenance of access roads, site maintenance and waste hauling services. Although not crucial, these factors can create problems and increase operating costs. Proximity to a population centre is desirable.

## Utilities and Fuel

The plant will require boiler fuel, cooling water, process water, electricity, potable water and fire water. However, these needs should be easily satisfied close to a population centre.

## Environmental Considerations

The facility must be designed to conform with all prevailing Provincial, Federal and local environmental regulations. It will, however, generate significant volumes of highly polluted wastes. If located in a major centre, the waste treatment system may not be required. Alternatively, waste treatment will be required and access to a stream is desirable for discharge of treated effluents.

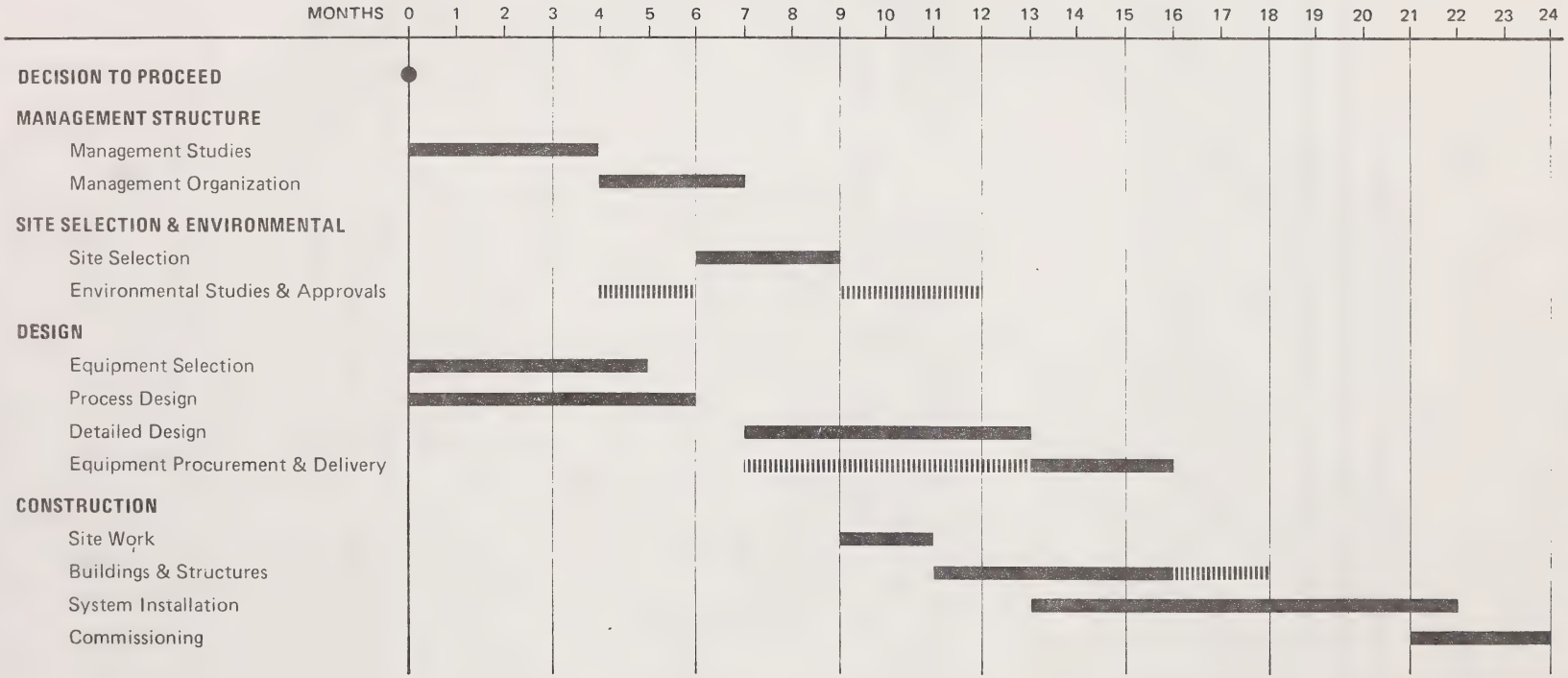
Although scrubbers will be provided for air pollution control, it is desirable to locate the facility downwind of the residential districts. Location in a heavy industrial area in a large industrial and research centre should be quite acceptable from an environmental standpoint.

## Special Criteria

As a major function of the proposed facility is research and development into the technologies for conversion of poplar to ethanol, a special site selection criterion should be the proximity to a major population centre to facilitate personnel and information exchange. A number of research personnel may regularly travel to this facility to work for a specific period of time. Similarly, the plant personnel may be involved in work in other research laboratories. Quick access to technical information will affect the overall operational efficiency.

## Conclusions

Because of the research and developmental function of the proposed facility, the most significant site selection criterion is proximity to or good accessibility from a major industrial and research centre. Normal considerations for industrial facilities such as utilities, services, and proximity to raw materials and market, should be considered, but are not overriding concerns. Good "quality of life" is desirable in view of the nature and skills of personnel that the proposed facility hopes to attract.




PROGRAM IMPLEMENTATION SCHEDULE

FIGURE 13

IMPLEMENTATION SCHEDULE

An 24-month Program Implementation Schedule, based on simultaneous start of management, environmental and engineering work, is presented in Figure 18. Primary program components are:

1. Management Structure
2. Site Selection and Environmental Studies
3. Design
4. Construction

The first step is the definition of a management organization by commissioning a detailed management study. Site selection, and process design activities could be initiated parallel to this study if time were an important element.

Process design and equipment testing are the most crucial next steps in the technical development of the program. Process and equipment testing was initiated during the study phase and should be continued during process design. Areas of priority are:

- Tests on substrate prepared with Wenger extruder.
- Pulp handling and dewatering equipment.
- Lignin handling and dewatering equipment.
- Continuous acid hydrolysis process.

The construction schedule assumes that the environmental assessment requirements are minimal because of the size of the facility and site work can start immediately once a site has been selected. Construction is based on sequential tendering to allow some design and construction overlap.

APPENDIX A

**TERMS OF REFERENCE**

# TERMS OF REFERENCE FOR A POPLAR CHEMICAL DEMONSTRATION PLANT AND SUPPORT FACILITIES

## Introduction

There is a sizeable underutilized poplar resource in Alberta some of which is suitable for the manufacture of conventional wood products such as lumber, veneer, waferboard and pulp, but a large volume is classified as unmerchantable based on the above mentioned uses. Studies commissioned by the Department of Energy & Natural Resources* as well as the Federal Government indicate that there are some very promising technologies being developed that could turn this unmerchantable poplar into valuable energy and chemical products.

Although not the sole reason, development of this resource base is the primary motive behind the Department's decision to take the initiative in piloting some of these novel technologies with the hope of eventually seeing them applied on a commercial scale.

One such promising field is that of converting wood to ethanol via acid or enzymatic hydrolysis using various process designs depending on what product or by-product is desired. Our strategy is to build an optimum sized poplar-based chemical demonstration plant and laboratory support facility with the capability and flexibility to technically evaluate th maximum number of process variables. In order to achieve the desired results, we require outside expertise to provide the conceptual design, recommend the optimum scale, estimate capital costs, select a suitable site and suggest project and construction management options as well as plant operating and management options available to the Department.

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* Hereinafter referred to as the "Department"

Phase I - Pre-engineering Study

Purpose

To identify, describe and evaluate the equipment and design configurations required in all acid and enzymatic-based wood to ethanol processes and pretreatment methods for the purpose of selecting compatible components and incorporating them in the design of an optimum sized pilot plant and support facilities with the versatility to technically evaluate the maximum number of known process variables using 100% poplar feedstock. To recommend the appropriate plant size and produce flow sheets and block diagrams of the complete facility along with estimated capital costs. To recommend a project management and construction team and provide plant operating and management options.

Guidelines for Consultant

- All equipment and plant design to be based on 100% poplar feedstock.
- Scale of pilot plant must be greater than 1 tpd of oven dry feedstock and no larger than 50 tpd.
- Facility must contain appropriate laboratory support facilities and sufficient instrumentation to ensure adequate monitoring and evaluation of process variables.
- Entire facility must not cost more than 25 million dollars.
- Facility to be designed to evaluate primarily enzymatic hydrolysis but design should allow for evaluation of acid hydrolysis as well.
- Plant design must allow for optimum flexibility in process evaluation and equipment change in order to facilitate analysis of future breakthroughs, advances and developments in this technology.

- Plant must be designed to evaluate a minimum of three separate pretreatment processes (Iotech, Stake and Gulf) and allow space for installation of at least one more process.

- If at all practical, consideration should be given to allow for continuous processing in order to simulate commercial operating conditions.

- Design to include provisions for feedstock preparation, storage and infeed, pretreatment, separation of components (lignin, hemicellulose, cellulose) prior to hydrolysis, acid and enzymatic hydrolysis, simultaneous saccharification and fermentation, separation of lignin and hemicellulose after hydrolysis and fermentation, distillation, product storage/disposal, enzyme production, laboratory and office space.

Consultant's Obligations

- To provide a description of processes evaluated.

- To provide flow sheets of the conceptual process design(s) and block diagrams of complete facility.

- To recommend the optimum plant size following guidelines set down.

- To provide approximate cost estimates envisioned to include a list of the basic elements of a plant facility and offshoot equipment required to supplement main process line or lines. Cost estimates should cover land requirements, buildings, equipment, manpower, services and operating costs.

- To provide a sensitivity analysis of equipment costs relative to their capacity.

- To determine and verify the patent position of all process and equipment developers as well as to confirm their degree of cooperation and participation in the pilot plant facility.

- To recommend who should handle the engineering design, construction and project management.

- To explore the management and operating options available to the Department and recommend one.

- To identify and confirm uses and outlets for pilot plant products and by-products whether for research or commercial purposes. To suggest a means of disposal should no outlets exist.

- To identify the extent and degree of activity required in R, D & D relating to deveopment of processes and further processing of pilot plant products and by-products.

APPENDIX B

# TECHNOLOGY REVIEW

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INTRODUCTION

An extensive literature survey has been performed to determine the extent of work being done in ethanol production from cellulose. This survey has been supplemented by discussions with researchers, process engineers, plant operators, and by visits to locations where work is being performed in crucial areas of cellulose conversion.

Results of our findings, with assessments and preliminary selection of technologies are presented in Appendix B under the following headings:

- Pretreatment
- Acid Hydrolysis
- Enzymatic Hydrolysis
- Bacterial Conversion of Cellulose to Ethanol
- Fermentation
- Distillation - Ethanol Separation
- Pentose Fermentation
- By-Products

## Pretreatment Processes

For the production of ethanol from sugars by fermentation, no pretreatment is required. Similarly, processes for saccharification of starches are well established. However, lignocellulosic materials are relatively difficult to hydrolyze. The difficulty arises from two sources:

1. The nature of the lignocellulosic bond, which protects cellulose molecules.

2. The tight crystal structure of cellulose, which prevents the entry of hydrolysing agents to the critical bonds between the glucose units in cellulose.

While not all cellulose hydrolysis processes include a "pretreatment" step to increase the availability and susceptibility of cellulose to hydrolysis, the most promising processes include such a step. These pretreatments include:

- Autohydrolysis, which is a steam treatment at 190°C to 250°C for periods of 20 minutes to less than one minute. This is generally followed by explosive decompression to break the lignocellulosic bond.

- Mechanical treatments such as 2-roll milling.

- Treatments with dilute acids which dissolve hemicelluloses.

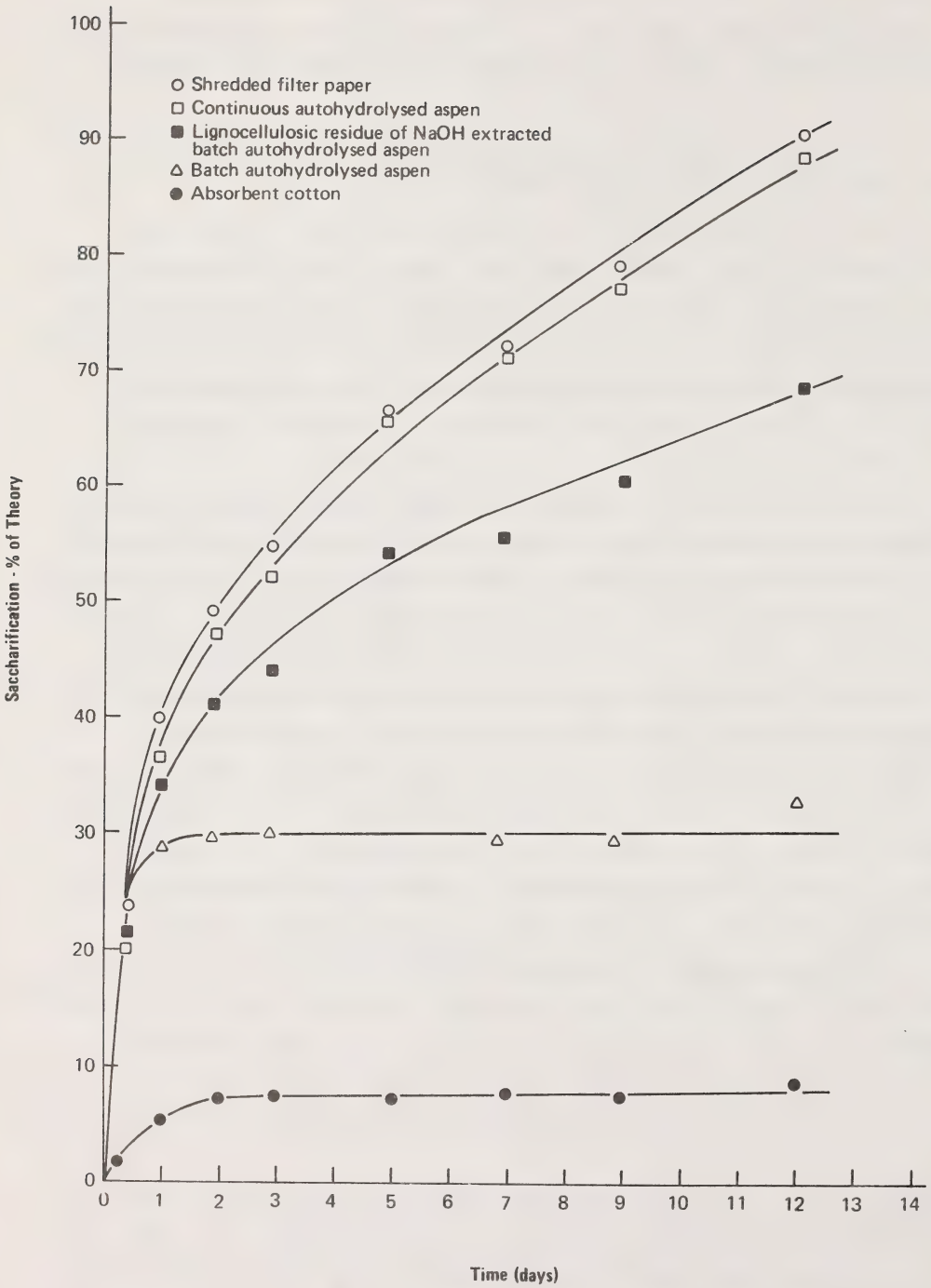
- Treatments which swell or dissolve the cellulose, usually by strong alkali or strongly alkaline metal solutions or by strong acids, especially hydrochloric or hydrofluoric acids.

Biological pretreatment involving fungal degradation of lignin is also under study. An extensive report by Battelle Columbus Laboratories in March 1980[3] reviewed pretreatment methods in 176 pages and 350

references. Even in the short time since that report was written, significant progress has been made in a number of research laboratories on pretreatment methods. Those most relevant to this study are discussed here.

## Autohydrolysis and Explosive Decompression

The term autohydrolysis refers to the fact that on heating with steam, wood species such as poplar generate acids which catalyze the hydrolysis of the hemicellulose of the wood and break the lignin-carbohydrate bonds. Such a steam treatment, under well chosen conditions of time and temperature, hydrolyse the hemicellulose almost completely, freeing the monomeric sugars, mainly glucose and xylose. At the same time the lignin-carbohydrate bonds in the wood are broken and the lignin becomes alkali soluble. Whether or not the hemicellulose and lignin are removed following autohydrolysis and whether or not autohydrolysis is terminated by explosive decompression. The cellulose following autohydrolysis is far more susceptible to acid or enzyme hydrolysis than the untreated wood. This susceptibility is demonstrated in Figure B-1, which compares the rates of enzymatic hydrolysis of various raw materials[4]. The optimal conditions for lignin solubility and for susceptibility to hydrolysis have been defined in several papers from Wayman's Laboratory at the University of Toronto (4,5,6). It was shown that if autohydrolysis is prolonged beyond the optimum, lignin recondenses, partly with itself and partly with hemicellulose components, and the resulting product loses some of its value. Explosive decompression does not affect the chemical results, but it does convert the cellulose to a relatively fine powder, making it easier to process.




ENZYMATIC HYDROLYSIS OF VARIOUS SUBSTRATES BY CELLULASE, 10.0% SUBSTRATE, pH 4.8, 50°C

FIGURE B-1

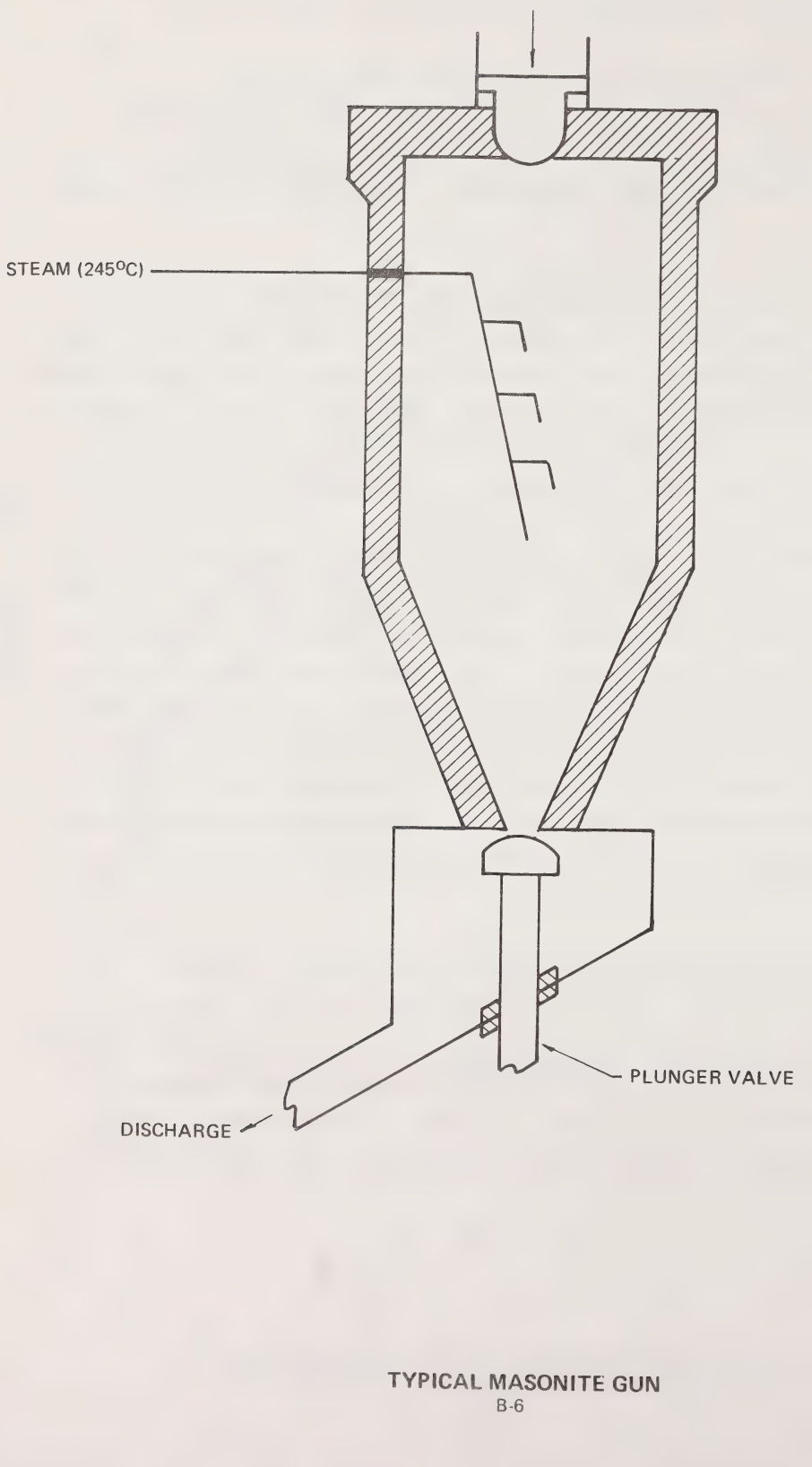
There are several items of equipment available for autohydrolysis, including:

- the continuous reactor devised by Stake Technology Ltd.
- the rapid cycle batch reactor (Masonite Gun) employed by Iotech Corporation,
- the extruder-cooker marketed by Wenger Manufacturing Inc., of Sabetha, Kansas.

All three of these are well-established items of equipment. The Stake reactor is operated at 5 to 10 tonnes per hour in 3 wood-based cattle feed plants; the Iotech Masonite Gun is standard equipment in all Masonite board plants, and the Wenger extruder-cooker has been used for about 35 years for grain processing, although its adaptation to cellulosics is rather new. Sketches of this equipment are presented in Figures B-2, B-3, and B-4.

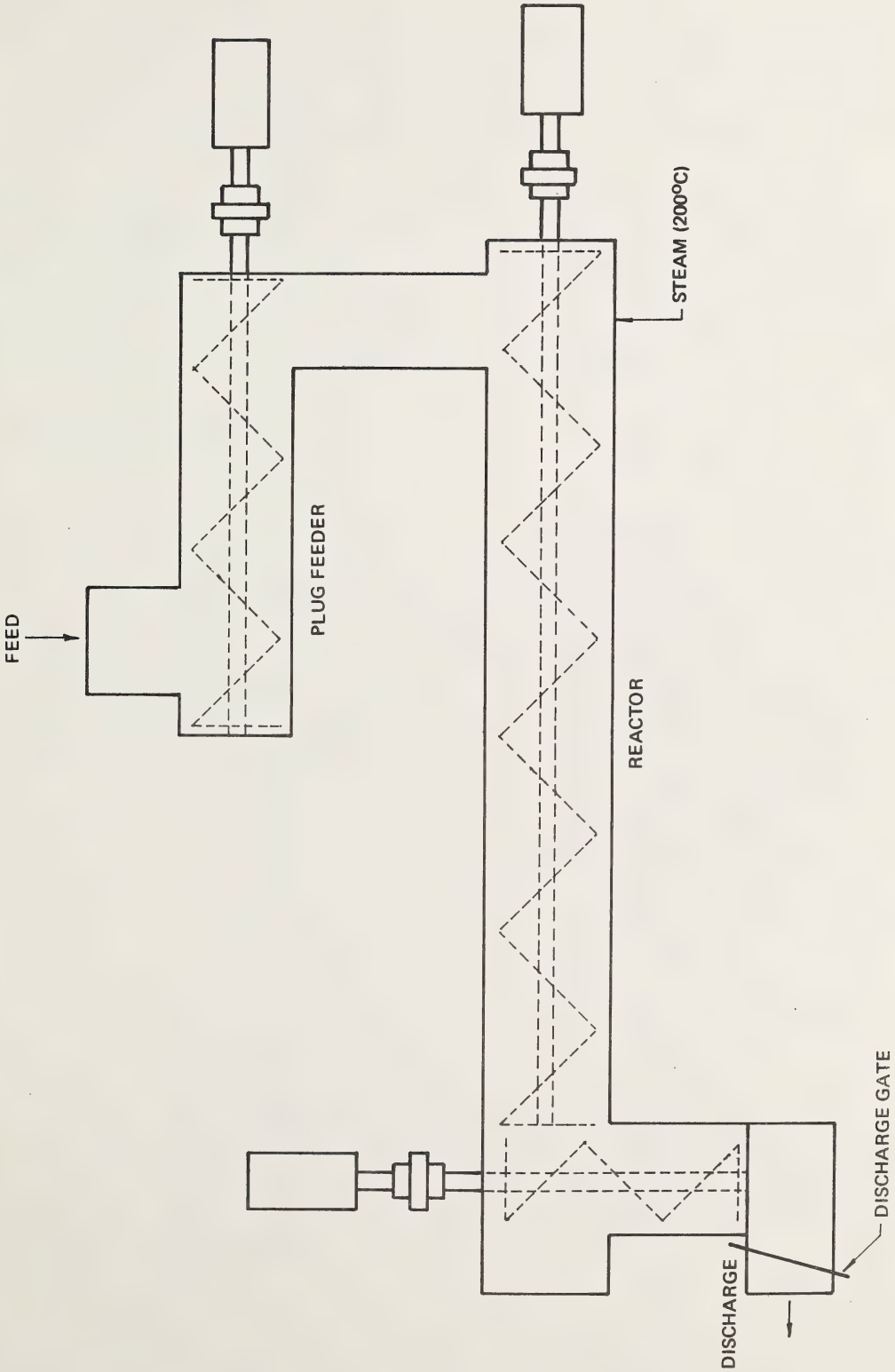
Generally, the Stake equipment autohydrolyses wood at about 200°C for 3 to 6 minutes. The Iotech equipment is operated at about 245°C for about one minute, for a total cycle of about two minutes. There are no obvious major advantages associated with either of these items of equipment; it could be expected that less maintenance would be needed with the Iotech equipment, which has no moving parts. The major advantage of the Wenger equipment is that no steam is used in the process, heat and pressure being generated by internal friction of a helix against a die. This could represent a major saving by eliminating the capital and operating costs of a high pressure boiler.

The above review presents the main features of the most accepted pretreatment processes. In practice, conditions should be chosen to minimize furfural formation during autohydrolysis (7) by acid reaction with xylose, since furfural may interfere with fermentation and enzyme action. Reaction conditions also influence the degree of dissociation of the lignin-cellulose bond and susceptibility of lignin to alkali extraction.



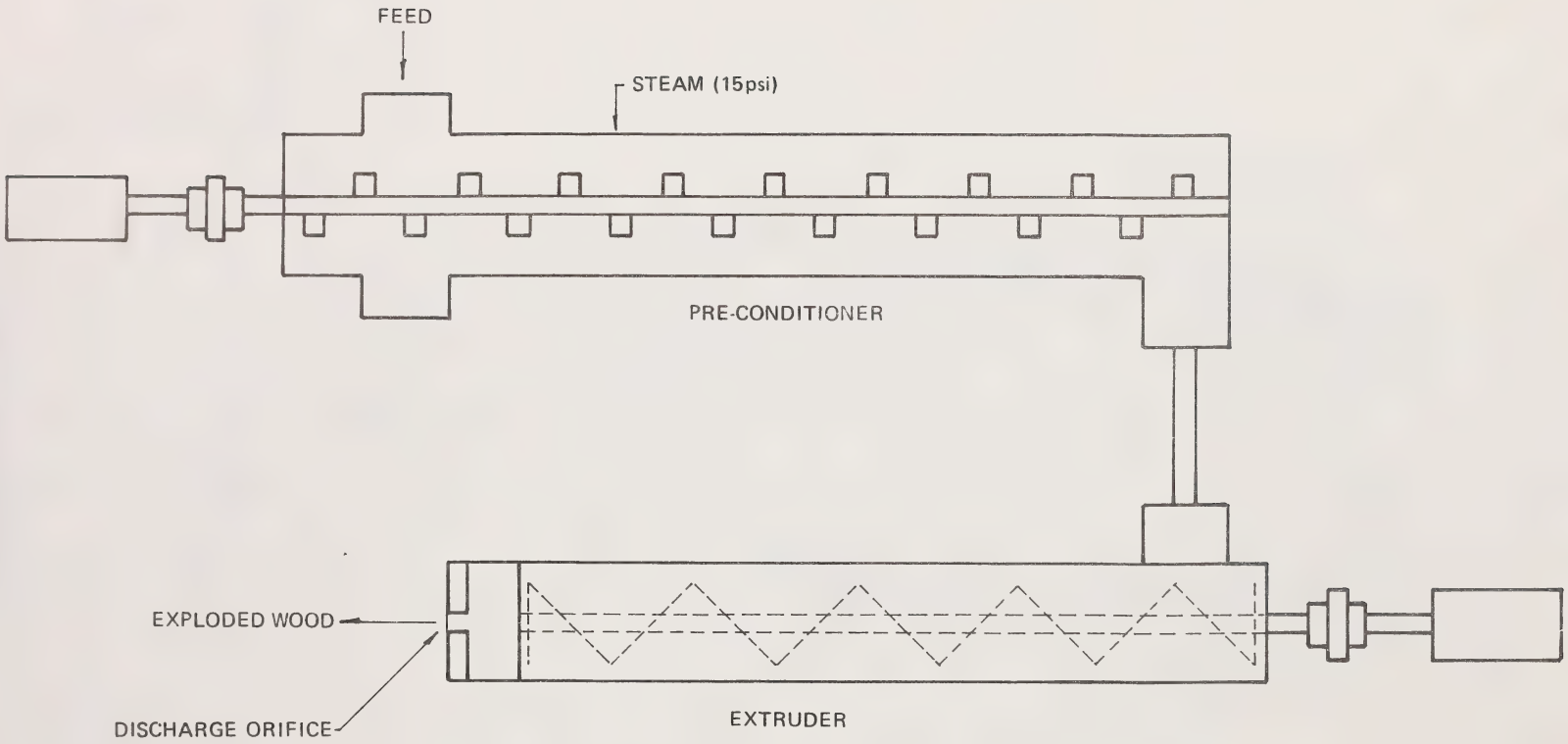

TYPICAL MASONITE GUN

FIGURE B-2




STAKE AUTOHYDROLYSER

FIGURE B-3




WENGER EXTRUDER

FIGURE B-4

The effect of a catalyst on autohydrolysis has been studied by several groups, especially by The General Electric Company (GE) (8). One of the most frequently mentioned catalysts is sulphur dioxide. This should be borne in mind when considering the metallurgy of such reactors.

It has been estimated that the cost of steam explosion pretreatment is $8/tonne BDWE including both capital and operating costs. This is equivalent to 2.5¢/litre of ethanol. While such optimistic figures can be questioned, there is little doubt that this is a relatively inexpensive and effective process for preparing wood chips for hydrolysis.

## Mechanical Treatments

The United States Army Development Centre at Natick, Massachusetts has studied and reported on mechanical compression milling in a 2-roll mill as a pretreatment for some cellulosics, particularly for waste paper. They have shown that such mechanical action increases the susceptibility of waste paper or poplar shavings, to enzyme saccharification. This is attributed to breaking down the cellulose structure and improving access to the critical bonds. In Natick's laboratory, 2-roll-milled poplar and autohydrolysed poplar gave about the same results in trials about four years ago. There are, however, some rather serious problems with the 2-roll mill. It is quite difficult, if not impossible, to get uniform treatment and the capacity of the equipment is much reduced by the need for repeated passes. In their most recent report (9), they need 15 passes through the rolls to achieve modest pretreatment. By passing newsprint through a 4-roll mill, they achieved similar results in only six passes, using 0.46 kWh/kg of newsprint.

Katzen (10) has claimed development of a pretreatment process by the University of Arkansas Biomass Energy which is based on combined mechanical-chemical pretreatment. Energy consumption is claimed to 200 kWh per tonne of BDWE. However, patents are pending and no process details are available for assessment.

## Organosolv Pulping and Saccharification

There are at least three and possibly five processes for removing lignin from wood by means of organic solvents. These processes also serve as pretreatments ahead of saccharification of the delignified cellulose. The earliest is the ethanol pulping process of Kleinert, formerly of the Pulp and Paper Research Institute of Canada (11). The butanol extraction process of Pye and Humphrey (12) is to be considered seriously, especially if the sugar solutions derived from poplar hydrolysis are to be used for butanol manufacture, a significant possibility outside the scope of this report. April (13) has also reported on butanol organosolv pulping. Dr. Laszlo Paszner, at the University of British Columbia, has still another system (see site visit report, Appendix B).

The organosolv processes are based on the concept that lignin is sufficiently soluble in organic solvents at high temperatures that its partial removal enables the fibers of the woody material to be separated from each other with low energy input. In a plant designed to make ethanol or butanol, these would obviously be the solvents of choice and they have been investigated thoroughly along with many others. Attempts have been made to catalyse organosolv pulping (13) but generally these have not been successful. It was hoped that these solvents would be rather specific, dissolving the lignin and leaving the hemicellulose (high yields of fermentable sugars) but these hopes have also not been realized. Yields in organosolv pulping are no better than in conventional forms of pulping such as kraft or sulphite.

Success depends also upon the efficiency of solvent recovery and energy consumption. Katzen (11) and Diebold (14) have described the ethanol process in some detail, including cost estimates. Wood chips are extracted in three stages by aqueous ethanol at 200°C. The lignin is recovered by evaporation and precipitation. The processing cost for cellulose

preparation is said to be 7¢/kg of wood, which would be equivalent to 14¢/kg cellulose or 30¢/litre of ethanol as pretreatment cost. This excludes the cost of the wood, capital cost and all further processing costs such as saccharification, fermentation and distillation. This is very high, about ten times the cost of autohydrolysis. The butanol pulping processes are no better economically.

Pazner claims major improvements to the conventional organosolv pulping processes by using a solvent along with an acid catalyst to extract lignin and to hydrolyze cellulose simultaneously. The solvent prevents sugar decomposition, resulting in up to 95% hydrolysis and almost complete recovery of high quality lignin. However, work has only been performed on a laboratory scale and continuous bench-scale tests are required to establish the technical and economic feasibility.

Although the economics of organosolv pulping are not, so far, favourable, the concept is an attractive one and one can hope for improvement as studies continue. Simultaneous lignin removal and high yield saccharification at high sugar concentration should improve the economics significantly, but pilot-scale testing and detailed economic analyses are required.

## Swelling and Solubilization of Cellulose

Since the cellulose crystal structure is a major handicap to cellulose saccharification, it would be expected that dissolution of cellulose, which would disrupt the crystal structure, would be an effective pretreatment for saccharification. Generally, cellulose solvents fall into two groups, concentrated acids (41% hydrochloric acid, 85% phosphoric acid and 72% sulphuric acid) and solutions of metals in ammonia, organic amines or alkalis (cuprammonium, cupriethylene diamine, cadmium ethylene diamine (cadoxen), alkaline ferric tartrate).

It should be noted that complete solution is not necessary as a pretreatment stage. Any substantial degree of swelling of the cellulose will open its crystal structure to permit more rapid saccharification. Suitable swelling agents include 10 to 18% sodium hydroxide solutions.

The main proponents of cellulose solution pretreatments are the Purdue Group under Tsao and Ladisch (15). At first, they favoured cadoxen or the ferric tartrate as pretreatment and then obtained better results with 70 to 80% sulphuric acid. Their process now consists of a prehydrolysis with dilute sulphuric acid and removal of the hemicellulose, followed by cellulose dissolution in strong sulphuric acid. Methanol is added to the solution to precipitate the decrystallized cellulose, which is then suitable for hydrolysis. However, this process has not been tried on any scale above the laboratory and is not considered to be economically attractive.

Other Acid Pretreatments

The concept of a necessary pretreatment before saccharification of wood is generally accepted. In many processes, however, the pretreatment and saccharification are closely linked. The best example is the New Zealand process, developed for pine (16). In this process, pretreatment is carried out with dilute sulphuric acid at 150°C, a temperature at which, they claim, the sugars are not harmed. The acid used for pretreatment is the percolate, a filtrate from the saccharification state, which uses fresh dilute sulphuric acid at 190°C. In other words, there is a countercurrent flow in which fresh pine wood is pretreated at 150°C with acid sugar solution from saccharification. The solution from the pretreatment is fermented. While the New Zealand process gives good yields with pine, it is probable that for poplar, a 3-stage process would be better: autohydrolysis, followed by the two stages of the New Zealand process.

In an ideal process, both hemicellulose and lignin would be removed following autohydrolysis and ahead of saccharification. This is now being studied by the New Zealand group. A demonstration plant for ethanol from pine is planned for New Zealand.

It is reported that the Forest Products Laboratory at Madison has modified their original single stage dilute sulphuric acid hydrolysis process to a 2-stage process, to incorporate dilute acid pretreatment (17). The details of this new Madison process are discussed under the Acid Hydrolysis section.

Both dilute and strong acid pretreatments have also been employed ahead of strong acid saccharification. In the German processes, Rheinau-Bergius and Udic-Rheinau, both of which employed 41 percent hydrochloric acid at 20°C, there was a dilute acid pretreatment, 1 percent hydrochloric acid at 130°C in the former process and 32 percent hydrochloric acid at 20°C in the latter. The Japanese process by Noguchi-Chisso uses 5 percent hydrochloric acid at 100°C as a pretreatment ahead of an HCl-gas saccharification. In another Japanese process, by Hokkaido, 1.2 to 1.5 percent sulphuric acid is employed in pretreatment at 140°C, followed by saccharification using 80 percent sulphuric acid.
Both the German and the Japanese processes mentioned here have been operated on a semi-commercial or pilot plant scale -- the original Bergius process, with no pretreatment, having been operated on the full commercial scale.

## Other Pretreatment Processes

The contrast between the bewildering array of processes invented and developed to the pilot scale and the very few processes commercialized is testimony to the great difficulty of cellulose hydrolysis. Some other approaches to pretreatment which are still being pursued in the laboratory follow.

Several laboratories, including Kirk at the Forest Products Laboratory, Madison, Wisconsin (18), are studying microbiological degradation of lignin as a pretreatment for cellulose hydrolysis. If such a process were to attack the lignin only and leave both cellulose and hemicellulose intact, higher yields of ethanol could be expected. While much has been learned in these studies about the mechanism of microbial lignin degradation, there has been as yet no practical issue, nor does there appear to be much practical prospect.

Radiation has been used to weaken the cellulose structure to induce susceptibility to hydrolysis (19). The amounts of energy input for any detectable effect have been beyond any practicability.

Thermal treatments during laboratory milling have also been studied (20), using fir at 200°C during 40 minutes of milling as a pretreatment. When followed by acid hydrolysis, the results suggested complete conversion of cellulose to glucose. Hot milling as a process is well established in the plastics industry, but there has been no follow-up to this 1969 report.

Pyrolysis of pure cellulose carried out under carefully controlled conditions may serve as a pretreatment for acid saccharification (40), yields of about 70 percent being achieved. Vacuum pyrolysis was particularly effective, forming a tar which, when hydrolyzed with sulphuric acid at 97°C for 2-1/2 hours, yielded 67 to 79 percent glucose. In applying these results to cottonwood, it was found that pretreatment and

removal of hemicellulose was required in order to obtain good yields of glucose. These heat treatments have been carried out only in the laboratory. If a "conventional" pretreatment is required, pyrolysis would represent an additional step ahead of acid hydrolysis, and it is difficult to assess what advantage, if any, it would offer.

Tests are being conducted (21) at Ontario Research Foundation (ORF) on autohydrolysis of corn stover, using wet oxidation techniques, although oxygen is not injected. Up to 60-70 percent hemicellulose hydrolysis has been achieved with good cellulose liberation. However, the stover must first be finely ground to increase reaction rates. The potential advantage is good heat recovery, although capital costs are expected to be high.

## Acid Hydrolysis

The objective of hydrolysis is to convert the carbohydrate components of the wood to sugars. The amount of sugar formed is the measure of the effectiveness of the hydrolysis. Acid hydrolysis is the method historically used for the production of ethanol from wood. This can be broken down into four processes:

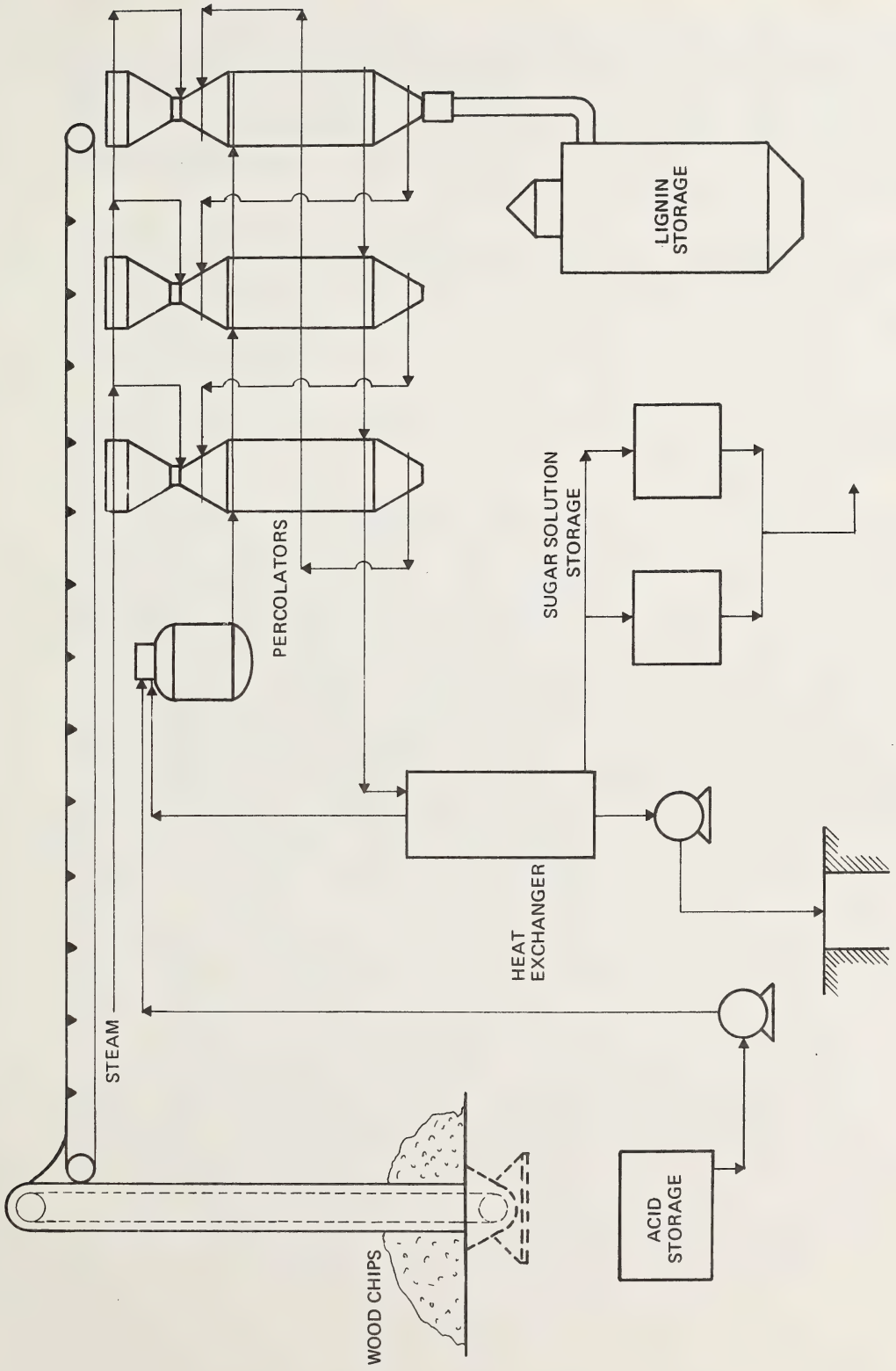
- Single Stage High Temperature, Dilute Acid
- Strong Acid
- Multi-stage Dilute Acid
- Dilute Acid with Solids Recycle.

## Single Stage High Temperature, Dilute Acid

There are several such processes, the Scholler and the Madison processes (dilute sulphuric acid at 170 to 180°C) and the Dartmouth and the New York University processes (dilute sulphuric acid at 245°C for very short times). The Scholler process, developed and operated in Germany in World War I, is the basis for about 30 operating wood hydrolysis plants in the USSR making about 50 million litres per year of ethanol. The Madison process was installed at Springfield, Oregon, during the Second World War but never operated at more than half the design capacity. These two processes represent the sum total of full scale experience in making ethanol from wood, and in neither case do the circumstances or available data lend themselves to acceptable economic analysis, or proper engineering analysis.

The main criticisms directed against the Scholler and Madison processes are the low yield of glucose, about 60 to 70 percent of theory or less, and the low glucose concentrations, 4 to 5 percent, which increased ethanol distillation costs.

Figure B-5 shows the Scholler process. Wood chips are fed to a series of vertical percolators which are pressure vessels, ceramic-lined, operated batchwise. The temperature is maintained at 170°C for 2 to 4 hours. Sulphuric acid at 0.4 percent concentration is added to each percolator and the sugar solution withdrawn as it is formed. It should be emphasized that glucose is unstable in acid solutions at these temperatures and the rapid removal and cooling of the percolate is important to preserve the products of hydrolysis. About 6 parts of acid solution are used per part of wood, and the resulting solution contains approximately 4 percent glucose. Yields of about 50 percent of theory are obtained. Lignin is recovered from the bottom of the percolator as a char, useful primarily as fuel.




THE SCHOLLER-TORNESCH WOOD HYDROLYSIS PROCESS FIGURE B-5

The Madison process used 0.5 percent sulphuric acid at an acid solution/ wood ratio of about 10/1, and a temperature of 180°C. Yields were again about 50%. The plant was designed for semi-continuous operation, which included reuse of sulphuric acid solutions with low sugar concentrations. Average sugar concentration was about 5%.

This is a rather poor record and poor performance to draw upon for the design of future wood-based alcohol plants. Nevertheless, in the newly scheduled Brazilian wood-based alcohol plants, the same basic design is being followed. In practice, however, multi-stage plants are more likely to be installed, with one or two stages of pretreatment before dilute hot sulphuric acid hydrolysis.

The work of Saeman in connection with the Oregon plant resulted in excellent kinetics studies, the best so far produced (22). His work was extended at Dartmouth by the group under Grethlein using a small laboratory continuous plug flow reactor. The Georgia Institute of Technology (GIT) group under Roberts (23) has also recently contributed to the kinetics of acid hydrolysis, using very small glass batch reactors (see Appendix C). These new studies confirm that at higher temperatures and shorter times, yields should be better than those obtained in the earlier processes. At temperatures of about 240°C yields over 60 percent of glucose, compared to earlier values of 50%, are predicted if reaction times can be kept to about 3 seconds (24). A serious attempt has been made to realize Grethlein's contribution by Rugg, of New York University. Using an adapted Werner and Pfleiderer twin screw extruder, sawdust has been steamed to 245°C and sulphuric acid added just ahead of discharge. Reaction time is estimated at 10 seconds. The resulting dark brown to black paste contains glucose at something just under 50% yield (25). The New York University process is capable of limited further improvement, especially by shortening reaction time and probably even more by introducing autohydrolysis, as has been found by Grethlein's group. The latter has now found (24) that chips held in a digester for one minute with steam at 5500 kPa, 270°C and suddenly

released to atmosphere, undergo complete separation into fine particles and the hemicellulose is completely hydrolysed and can be washed from the cellulose. Results of hydrolysis of this autohydrolysed feedstock have not yet been reported.

While these studies can be regarded as incomplete, the Werner & Pfleiderer Corporation is offering their system for large-scale ethanol plants.

## Strong Acid Hydrolysis

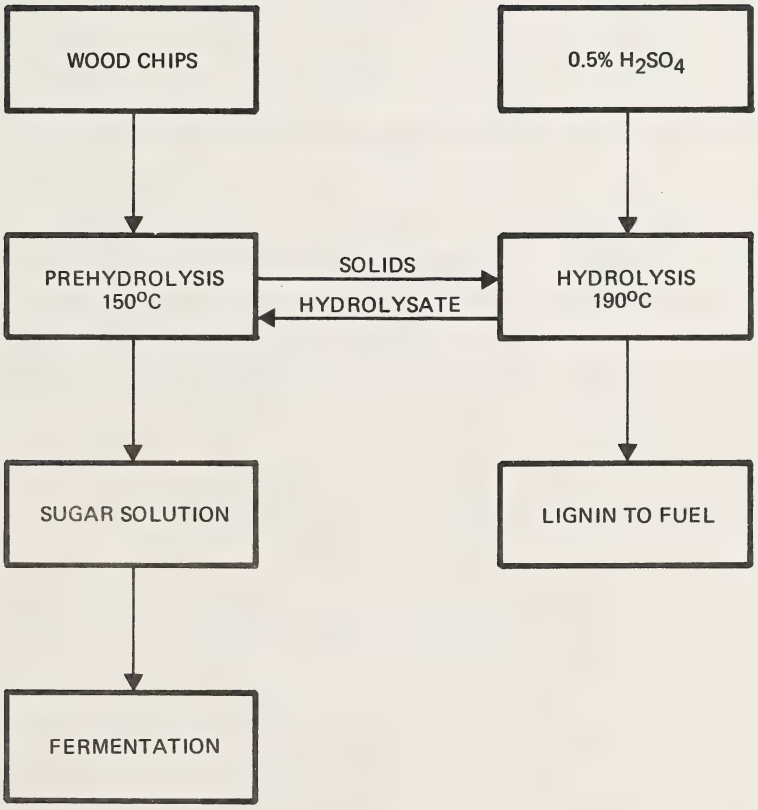
The use of concentrated hydrochloric acid as a hydrolysing agent goes back many years. The process is based on a finding of Willstatter about 1905 that when ordinary concentrated hydrochloric acid, which is about 37 percent HCl is fortified with gaseous HCl to above 41 percent, it becomes a cellulose solvent. It is a low temperature process and was developed on a commercial scale in Germany during World War I as a source of fermentable sugar, using beech wood. The process was described in detail by Bergius in 1937, including equipment, HCl recovery, products such as crystalline sugars, etc. (27). A few years ago, a laboratory was formed in Geneva, Switzerland, to study the cold, concentrated hydrochloric acid saccharification of cellulose, by a consortium of several clients led by Nestle and managed by Battelle, Switzerland. One great advantage of the process is that metallurgy was no problem since plastic reactors can be employed. A continuous process was devised in which 33 to 36 percent hydrochloric acid was used as a pretreatment at 20°C, followed by saccharification at 30°C using 41% hydrochloric acid fortified by HCl gas. This was practised on a pilot plant scale. About a year ago, the work was discontinued and as far as we know, the results have not been made public. Perhaps, like so many other processes, it foundered on the rock of "economics." Yet Bergius gave explicit directons and this process may yet have a future.

A Japanese process proposed by Hokkaido uses 80 percent sulphuric acid for saccharification of cellulose, pretreated by 1.2 to 1.5 percent sulphuric acid at 40°C.

## Multistage Dilute Acid Hydrolysis

It is now becoming clear that "cellulose" is by no means uniform, that in any given sample, some cellulose is readily hydrolysable while other molecules are broken only with difficulty. It is also generally accepted that sugar destruction by dilute acids is minimal at temperatures about 150°C and severe at about 190°C. The two new procsses which illustrate these findings were those developed at the New Zealand Forest Products Research Institute (NZFPRI) and the TVA-Madison process. The latter was developed jointly by TVA and the U.S. Forest Products Laboratory at Madison. A pilot plant was built which differed from the Springfield process, in that it included a prehydrolysis stage in which a relatively mild acid hydrolysis served to remove the hemicellulose ahead of the main hydrolysis step. It is reported (26) that the U.S. Forest Products laboratory presently propose a 2-stage process. In the first stage, steam at 170°C will be used for 20 minutes and the hemicellulose sugars washed from the pulp. It is not clear if some sulphuric acid is used in this prehydrolysis. In the hydrolysis of the residual cellulose, 1% sulphuric acid is used for "a short time at high temperature." A special feature of the latest development is a relatively low liquid to solid ratio, 2/1. A Masonite gun is being considered for this reaction. It is expected that a 20 percent sugar solution will be obtained, although washing the residual lignin and cellulose will reduce the concentration somewhat. Yields are still unknown but in excess of 70 percent of theory is expected.

The NZFPRI process differs from the others in an interesting way. They employ 0.5 percent sulphuric acid at 190°C for cellulose hydrolysis, and the resulting sugar solution still containing sulphuric acid is used for prehydrolysis at 150°C. It is thus the prehydrolysis solution which is fed to the fermenter, rather than the hydrolysis solution. Figure B-6 captures the essence of the process (27). Yields of about 80 percent are claimed from pine wood.




NEW ZEALAND HYDROLYSIS PROCESS

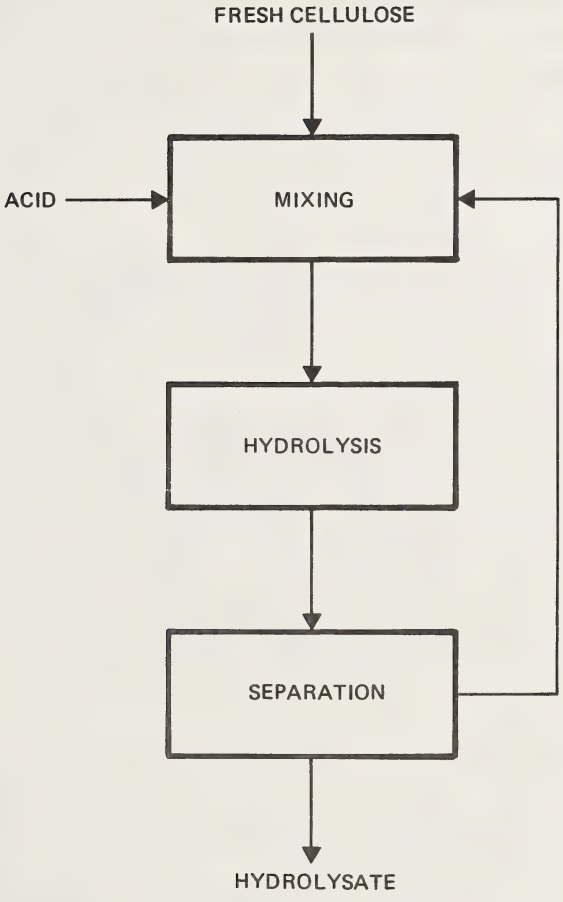
FIGURE B-6

These processes have been developed with conifers and the hardwoods, especially poplar, have yet to be investigated. However, it is beyond doubt that processing would be improved by autohydrolysis, and NZFPRI is now studying such a pretreatment.

A single reactor which simulates plug flow and carries out in a single vessel low temperature (140°C, 40 to 60 minutes) and high temperature (170 to 180°C, 150 to 170 minutes) dilute (0.6 percent) acid hydrolysis has been described recently by Inventa, of Switzerland (28). Sugar yields of 64 percent are claimed from debarked spruce, or 270 L/t of BDWE of pure ethanol. A 2-ton/day pilot plant is operating on this process. They estimate the cost of a 10-25 tonne/d of BDWE of poplar pilot plant at US $12 million for their process (29). This would translate into $17 million equivalent Canadians based on Wardrop's experience and does not include some of the expensive process systems such as pretreatment, enzyme production and hydrolysis, lignin and hemicellulose extraction. Also the degree of instrumentation, control and data logging is suited to a pilot facility rather than a facility capable of demonstrating processes under continuous operation.

Dilute Acid with Solid Recycle

If acid hydrolysis is carried out in five steps, starting with fresh acid in each step, very high glucose yields of up to 80 percent of theory are obtained (4). With such a high number of stages, one has what is in essence a solid recycle process. This process is shown in Figure B-7 (29). The work of Roberts at GIT has led him to the same conclusion. A paper by Pohjola (30) of Tampella, Finland, proposes a partial acid hydrolysis, separation of the sugar solution and recycle of the unreacted fibre along with fresh feed. This is a very promising process, three different laboratories in three different countries arriving at the same concept at the same time. However, such a process will have to incorporate pre-extraction of lignin to avoid inert solid build-up. A Stake type reactor has been proposed, but materials problems must be resolved. Depending on conversion per pass, achievable sugar concentrations may be quite low. Cost of handling and dewatering large amounts of pulp could be quite high.




CELLULOSE RECYCLE PROCESS

FIGURE B-7

## Enzyme Hydrolysis

A number of fungi attack cellulose -- the so-called "brown rot" fungi are familiar on decaying logs in any woods. The attack on cellulose is effected by a group of enzymes collectively known as cellulase. Since cellulose is non-homogeneous, several enzymes are involved. Some fungi are considered to produce a more or less complete cellulase, although this is, in our opinion, unlikely. The question of enzyme source and availability will be discussed further. At this time, attention will be drawn to the principal laboratory and pilot plant experiences with enzyme hydrolysis of cellulose.

There are a number of genuine advantages in enzyme hydrolysis of cellulose. The process takes place at ordinary warm water temperatures rather than very high temperatures. The action is specific and relatively gentle. The lignin is not attacked and may be recovered in useful form, relatively unchanged from the natural product in the wood. The hemicellulose is also largely unattacked although it may be degraded simultaneously to monomeric sugars by hemicellulases if desired. The reaction is, under the best circumstances, complete, that is 100% conversion of the cellulose is obtained with ease. The sugar solution so obtained needs no treatment of any sort to be ready for fermentation. There is no strong acid or toxic byproduct present to interfere with yeast action. In theory, it is an ideal process.

In practice, there are two key drawbacks: the cellulose in wood is not directly susceptible to enzyme attack, being protected by the hemicellulose and the lignin; and the quantity of enzyme and the lengthy time required for complete reaction affect the economics. The first of these problems has been solved, for all practical purposes, by the autohydrolysis pretreatment process and its variations. The second, the economic manufacture of an effective enzyme, remains to be solved. There is no operating enzyme-based cellulosic ethanol plant in the world today (in contrast, there are many enzyme-based starch, that is grain, ethanol plants), and the only large North American pilot plant, the Gulf plant at Pittsburgh, Kansas, is shut down for lack of support (see site visit report, Appendix C).

## Natick

The earliest enzyme saccharification work to gain wide recognition as having practical possibilities was that of the Natick, Massachusetts, United States Army Research Center. Their interest arose from a need to prevent the fungal deterioration of canvas webbing in military uniforms, and as a result Natick became interested in cellulase production and in the application of cellulase for ethanol production. The fungus identified by the Natick workers as the source of cellulase was Trichoderma viride, a green fungus improved by Dr. Elwyn Reese, of Natick, to the point where it has been renamed T. reesei. The history of Natick's work with cellulase is reviewed in a recent paper by D.D.Y. Ryu and Mary Mandels (31). This paper includes a description of the operation of their pilot plant.

The substrates originally chosen by Natick for conversion to alcohol were waste paper, including redundant government documents, and municipal solid waste, as well as cotton and sawdust. More recently, they have worked with poplar shavings. They have developed a pretreatment which includes milling, either in a 2-roll mill or a 4-roll mill. This has been described above under pretreatment. Many passes through the mill are required to achieve any degree of uniformity. There has been no adoption of this pretreatment process, in consequence of which their work has not resulted in any cellulose ethanol plant, even at a pilot plant level.

Their latest work has been concerned with improvement of Trichoderma strains for better enzyme yields. In this, they have had the cooperation of the Rutgers University Laboratory and the Lawrence Berkeley Laboratory of the University of California. Modest improvements in cellulase production have resulted, but these have not proven equal to the task. Also recently, Natick have turned their attention to a different microorganism, Aspergillus phoenicis, a bacterium, to make another necessary enzyme, beta-glucosidase, in recognition of the inadequacies of the Trichoderma cellulase.

Gulf

Enzyme hydrolysis of cellulose has also been a concern of Gulf Chemical Company, and G.H. Emert of the University of Arkansas utilizing the Kansas pilot plant built by Gulf Chemical. Emert has recently reviewed the history of his work on pilot scale conversion of cellulose to ethanol (32). Much of this work was carried on in cooperation with Wilke at Berkeley. In an effort to overcome the high cost of enzyme, Wilke developed a process step designed to recover and reuse at least part of the enzyme by recycling the enzyme sugar solution into contact with fresh cellulosic feed. This is supposed to bind the enzymes in the saccharification step effluent -- in the sugar solution -- to the unreacted incoming feedstock. There is no indication as yet how successful such a step has been.

The Gulf enzyme ethanol pilot plant was established to convert the cellulose content of municipal solid waste to alcohol. Its facilities include equipment for feed handling, for preparation of enzyme, for hydrolysis, for fermentation and for distillation. The plant, described in more detail in the site visit report (Appendix C), is capable of receiving one tonne of wood or other feedstock per day and converting this to ethanol. There are, however, certain critical missing parts: there is no provision for pretreatment nor is there a microbiological laboratory capable of preparing the fungus for inoculation into the cellulase production unit. For the past year, to the end of May 1981, the plant used as feedstock autohydrolysed aspen made in the Ottawa pilot plant of Iotech Corporation Limited. This high temperature [245°C short time (1 minute)] pretreatment was adequate to prepare the aspen for enzyme hydrolysis. There can be no doubt about the quality of the autohydrolysed wood delivered to the Gulf pilot plant. However, the absence of proper cellulase enzyme preparation facilities was a severe handicap. Cultures of the best strains of Trichoderma reesei were prepared by Wilke at Lawrence Berkeley Laboratory and shipped to Emert at Arkansas. Emert grew these up until he had a beer cask full of the green fungus, which he then shipped to the Gulf plant.

The material as received was highly non-uniform. At Gulf, the inoculum was added to fresh medium and grown to a high cell density, then water washed autohydrolysed aspen cellulose was added to induce cellulase formation. The results were highly variable, several days being required normally to produce enzyme. Then, even when the laboratory tests indicated good enzyme concentrations, and the enzyme solution was added to the autohydrolysed aspen in the hydrolysis tanks, the results again could not be depended upon, the time required for acceptable conversion to sugar solutions being sometimes several days or a week. In spite of this, high sugar concentrations about 12 percent, were obtained (33). By feeding fresh fibre into the hydrolysers as hydrolysis proceeded, high conversions per reactor volume were obtained. In the best run, saccharification yield of glucose was 88 percent, and 90 to 95 percent of theoretical yield of alcohol was achieved. The main problem with enzyme process, as practiced at the Gulf plant and probably with the process generally, is the complexity of the multiple enzymes which goes under the name of cellulase, and the unreliability of its preparations.

A major problem is the very high quantity of enzyme required to achieve a high degree of hydrolysis within an acceptable contact time. Lawrence Berkeley Laboratory and Natick, have proposed contacting the sugar solution with fresh fibre which, it is hoped, will absorb and reuse the enzyme. It would take about 90 percent enzyme recycle to solve the problem as it now stands. Stone and Marshall in their report (34) quote a recent reduction in enzyme cost, for in-house manufacture of enzyme, from 47¢/litre of ethanol to 9¢/litre. Similar improvements have been claimed by Katzen (17). This has not been confirmed by any data available to us, and is strongly doubted. But even if the lower figure were sustainable, it would cost as much as the wood.

Preparation of cellulase enzymes using anaerobic thermophilic bacteria have been reported by Khan (35) and Margaritus (36), however, the enzyme activities are low.

## Simultaneous Saccharification-Fermentation (SSF)

Another approach to enzyme hydrolysis has been to carry out the enzyme saccharification and fermentation simultaneously. The expectation has been that the removal by fermentation of the sugars as formed would reduce or eliminate product inhibition of the enzyme activity. This was a particular concern of Emert's (37). However, there are some disadvantages: in particular, the optimum temperatures for enzyme activity (50°C) and for fermentation (33°C) are far apart and a compromise is necessary. At this time, there is no apparent advantage in simultaneous saccharification-fermentation over the two separate steps.

## Plug Flow Enzyme Reactor

In a recent paper, Buchholz and his group at Dechema, Frankfurt, West Germany, have proposed a tubular enzyme reactor (38) instead of the usual batch (or continuous) stirred tank reactor. In their process, they propose to absorb the enzyme from the fermentation liquor by the autohydrolysed wood feedstock. With very little back-mixing, product inhibition should be minimized. They also claim high sugar concentrations. They give no details of their reactor, which may be simply a tube with a proper feed pump at one end, or may resemble the Stake reactor.

## Bacterial Conversion of Cellulose to Ethanol

There are bacteria which produce cellulase and which convert glucose to ethanol. Such bacteria should be capable of converting cellulose to ethanol in one step. This has been a central research activity of a group at Massachusetts Institute of Technology (MIT). They use a mixed bacterial culture including two species, Clostridium thermocellum and Clostridium thermosaccharolyticum. They have published several papers on this attractive process. Their latest results, reported in (39), describe new strains being developed which are good ethanol producers, without the simultaneous production of unwanted by-products such as acetic acid and lactic acid. Yields of ethanol based on cellulose feed are still low -- less than 50% -- and the time required is very long (6 days). These suggest that the process is still far from being ready to leave the laboratory.

One result of the MIT work which may be useful is the finding of a strain of C.thermosaccharolyticum which ferments xylose to ethanol. They report 0.43 g ethanol/g xylose. This is by far the highest ethanol yield from xylose yet reported. Since aspen contains a great deal of xylan, about 15 percent, this finding may have considerable practical importance in a process for conversion of aspen to ethanol.

The MIT process has also been studied by the General Electric Company (GE) using the same two organisms but with improved strains, isolated from soil samples in hot springs (40). G.E. reports progress with marked improvement in ethanol yields and faster growth rates. The G.E. work has an optimistic ring to it, but the data provided cannot yet lead to confidence that the process is ready for pilot scale operation.

Recently, much money has been spent on genetic engineering of yeast in order to confer on it the abilities exhibited by these bacteria, that is, the direct conversion of cellulose to ethanol. No encouraging reports have yet been issued.

## Fermentation

The technology for the fermentation of sugars from grain or wood is well established, although special considerations are involved in the latter case. Some developments in fermentation processes have been reported recently, and should be discussed. Fermentation discussions can be broken down into the following:

- Special Considerations for Wood Hydrolyzates
- Microorganisms
- Processes

### Special Considerations

Major considerations are:

- Concentration of sugar
- Toxicity
- Lignin separation
- Fermentation of pentoses

Most of the wood conversion processes produce low sugar concentrations in the hydrolyzate. This problem is carried through fermentation to distillation, resulting in very high steam requirements for ethanol stripping. An alternative is to evaporate the hydrolyzate before fermentation, although capital costs are high. With the advent of autohydrolysis, it appears possible to obtain high sugar concentrations (41), although problems related to handling of high consistency pulp must be solved.

Another major consideration in the fermentation of wood hydrolzates is the toxic nature of decomposition products of wood components. Although a large number of toxins have been identified, furfural and 5-hydroxymethylfurfural are the two major ones. Furfural is produced by the decomposition of pentose sugars and hydroxymethylfurfural from hexoses. Good control of autohydrolysis and hydrolysis processes is required to minimize their production. When present, their effect can be practically eliminated by maintaining a high concentration of active, acclimatized yeast culture during fermentation (42) as practised by some of the sulphite liquor fermentation facilities. Other methods of practical significance include: precipitation with lime; and adsorption on activated carbon.

For processes using autohydrolysis, special procedures must be used to separate lignin before fermentation to avoid solids build-up. These processes are discussed under by-product recovery.

Fermentation of pentoses, which form up to 25 percent of sugars in wood hydrolyzate, is also of interest. Rapid developments are taking place in this area and are reported below.

Microorganisms

Conventionally baker's yeast, Saccharomyces cerevisiae, is used for the fermentation of wood hydrolyzate. Other yeasts, which have the ability to assimilate other sugars present in the hydrolyzate, especially oligomers of glucose, are sometimes used. Among these are S. pombe or in the New Zealand work, S. guilliermondi. Whatever strain is used, it is very important to cultivate this strain in the plant laboratory to prepare the inocula, rather than simply to buy the yeast. When yeast is cultivated over a prolonged period on the wood sugars, it becomes adapted and its performance improved. New yeasts are being developed by means of genetic engineering to have enhanced fermentation properties for wood sugars (39).

In the past few years, a bacterium (Zymomonas mobilis) has been proposed by an Australian group in place of yeast for conversion of sugars to ethanol (43). It is claimed that fermentation rates are higher with higher fermentation productivity. With this bacterium, it is claimed, high concentrations of glucose (10 to 25 percent) can be rapidly converted to ethanol in yields up to 97 percent of theory. This is an active area of research, including Canadian studies which more or less confirm the Australian findings. These are still laboratory studies and it has yet to be shown that the laboratory advantages could be translated into industrial gains. This awaits further work.

## Processes

The following processes are being used, or can be used, for wood hydrolyzate fermentation:

- Batch
- Continuous
- Tower
- Vacuum
- Fermentation with Continuous Alcohol Stripping

The batch process has been historically used for grain alcohol production and is also being used for sulphite liquor fermentation at The Ontario Paper Co.'s Thorold facility. This system requires fresh yeast inoculation for each batch, resulting in high costs if large dosages are required. This is expected to be the case with wood hydrolyzate.

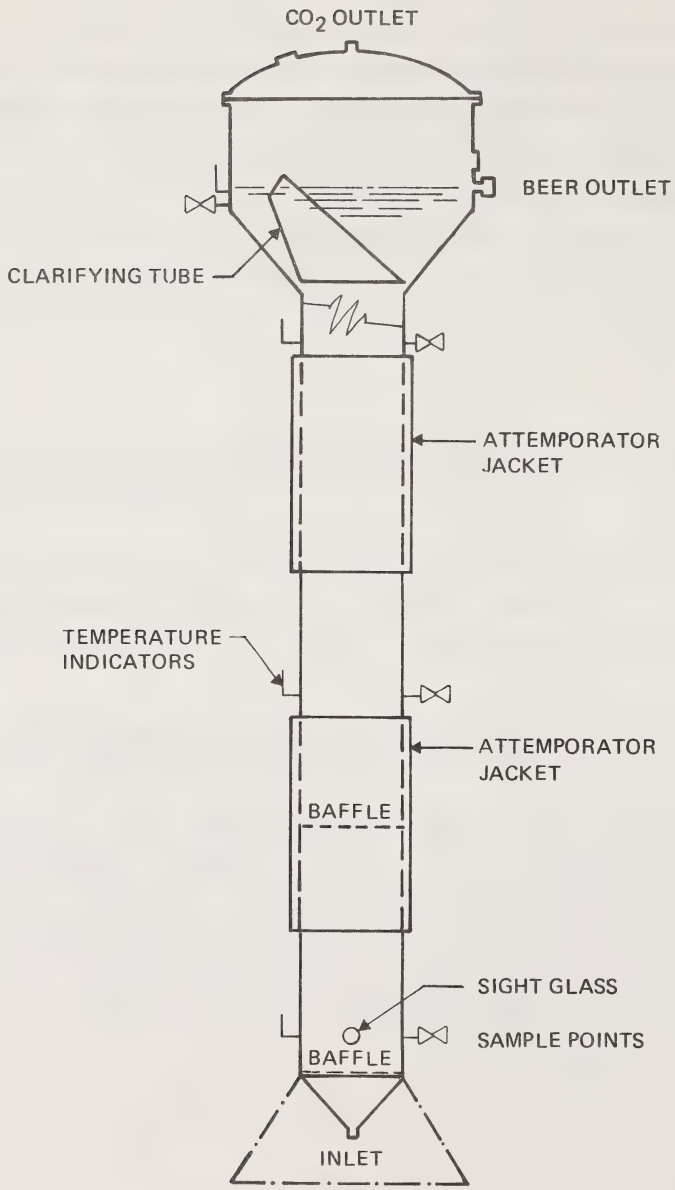
A continuous fermentation system without yeast recycle is being marketed in North America by Vogelbusch for grain alcohol production. For clear wood hydrolyzate, this process can be modified to incorporate yeast recycle. Such a process was provided at Canadian International Paper's Gatineau facility for sulphite liquor fermentation. Advantages are low yeast production cost, although centrifuges must be installed for yeast recycle. High yeast loadings appeared to control contamination well when the CIP plant was operated on sulphite liquor.

Tower fermenters have been used in the United Kingdom for commercial production of beer and vinegar. A typical sketch is presented in Figure B-8. Basic characteristics are:

- A cylindrical shape to permit plug-flow.
- A large settling zone on top to allow yeast retention.

It is reported that a continuous tower fermenter is feasible for ethanol production with a substantial increase in capacity. However, work with wood hydrolyzate will be required to select a suitable flocculating yeast.

Various means have been suggested for removing ethanol from the beer during fermentation, including vacuum distillation (the "Vacuferm" process) or the Alfa Laval Biostil in which hot streams from the still are used to strip ethanol from the fermenting brew. In such processes, fermentation and distillation are highly integrated. The primary objective is to conserve energy of distillation, as well as to utilize high sugar concentrations. It is expected that these processes will be initially developed and applied at the sugar and grain ethanol plants, and can then readily be adopted by the wood ethanol industry.




[Courtesy of O'Neil, et al (44)]

SKETCH OF THE APV TOWER FERMENTER

FIGURE B-8

## Distillation - Ethanol Separation

The technology for ethanol distillation is well established and need not be discussed in detail. No special considerations are required for wood-derived ethanol unless ethanol concentrations are very low. Proprietary systems are being offered by several companies such as Vogelbusch, ACR Corporation and Chemapec and are based on azeotropic or extractive distillation technology.

In simplest terms, distillation has two or three components: stripping the ethanol from the beer, rectifying the ethanol, that is, raising its concentration to 90 to 95 percent and finally, if desired for gasohol use, removing the last of the water by dehydration. A stripping column is a rather simple still designed to handle an unclear input flow and to be self-cleaning while steam evaporates the ethanol content of the beer. The vapours so produced are then fed to a rectifying column where ethanol is taken off overhead and water comes out the bottom. The ethanol so produced contains about 5 percent water and small quantities of other volatile organic compounds. For fuel use as a gasoline substitute, this product is quite satisfactory. For use as a gasoline extender (gasohol) or as an octane enhancer or for industrial ethanol, it needs to be purified and the last of the water and other compounds removed. This is normally accomplished in a second set of distillation columns in which an entrainer such as benzene or cyclohexane is added and the mixture distilled to produce anhydrous ethanol. These techniques are well established and widely practiced.

Some newer dehydration techniques involving water absorbents (the "Super Slurper" or cracked corn), molecular sieves or membrane separation have been proposed. These need not be considered at their present state of development since they will not affect the economy of the process significantly. Also if economically attractive, these will first be commercialized in sugar and grain based ethanol plants and could then be adopted by the wood ethanol industry.

## Pentose Fermentation

Poplar contains about 15 percent of a 5 carbon sugar, xylose (a "pentose") which, while being an excellent food for some yeasts, is not readily fermented to ethanol by ordinary yeasts. In practice, where wood sugars in sulphite liquor from hardwood pulping are fermented to ethanol, it is the 6 carbon sugars ("hexoses") such as glucose which become ethanol, while the pentoses remain in the solution. These may then serve as food for other yeasts, particularly Candida strains, which are grown for cattle feed. Such a process has been practiced for many years by the pulp mill at Attisholz, Switzerland. Some yeasts, including some Candida species can be induced to ferment pentoses to ethanol with rather modest yields, up to 50% of theory. Recently, Dr. Henry Schneider of National Research Council of Canada, Ottawa, has found that a yeast, Pachysolen tannophilus, will ferment xylose in high yields to ethanol in the presence of a small amount of air ("semi-aerobic") (45). This important finding is being pursued by NRC and it can be expected that this yeast, or perhaps some others, will become important in poplar ethanol production over the next few years. This is a "hot" topic of research at the moment.

R.J. Bothast and colleagues of the Northern Regional Research Center, U.S. Department of Agriculture, Peoria, Illinois, have reported (46) 80 percent yields of ethanol from 5 percent xylose solutions by their strains of Pachysolen tannophilus when fermenting at low pH, 2.5. This finding probably brings pentose fermentation into a practical range.

Other researchers, including George Tsao at Purdue, have adopted a different approach involving in essence an enzymatic conversion of xylose to xylulose, a sugar fermentable by ordinary yeast. The major problem with this approach is that the xylose  xylulose reaction is 85% in favour of xylose, resulting in very low xylulose yields. This problem could be overcome by simultaneous fermentation of xylulose, however, fermentation at low xylulose concentration proceeds slowly. A substantial amount of research is required before this process can be commercialized.

There are also fungi (Fusarium lini, an infection of flax) and bacteria (Clostridium thermosaccharolyticum) which convert xylose to ethanol. Reaction rates with F.Lini is very slow and work with Clostudium thermosaccharolytiaim is still at an early stage.

## By-Products

Of the various components of poplar, most of the glucose is derived from cellulose although some is obtained from hemicellulose. The remaining two major components, lignin and hemicelluloses, need to be extracted and converted to other by-products. In fact, it is crucial to the economics of ethanol from wood that valuable and marketable by-products be recovered from these components.

## Lignin Separation

There are several options for recovery of lignin in a process for ethanol from poplar wood. The lignin may be recovered following autohydrolysis, as proposed by Wayman (4), or following hydrolysis (47), or it may be carried through to distillation and recovered at the alcohol stripping column. The first two approaches are shown in Figures B-9a and B-9b. If the lignin is to be used for medical or chemical purposes, it is best to recover it following autohydrolysis. The procedure is to dissolve it from the fibre with dilute sodium hydroxide solution (0.5 to 3 percent NaOH, 70 to 100°C, 1 hour) and reprecipitate it by lowering the pH to 4.5. The lignin, separated by filtration or centrifugation, is then spray-dried to a fine tan coloured powder. The supernatant contains the hemicellulose sugars and may be fermented to ethanol or butanol or may be used to grow fodder yeast. This procedure, while apparently simple, is made more difficult by the small particle size of the lignin and the extra costs incurred need to be justified by the value of the product.

If an enzyme hydrolysis process is employed or if the lignin is to be used as fuel only in the case of acid hydrolysis, it may be carried through the hydrolysis and separated along with the fungus from the sugar solution by filtration or it may be retained through the fermentation and separated with the yeast. The procedure adopted will depend upon the process, the markets for the various products, and also upon whether or not enzyme or yeast recycle is practiced. For enzyme recycle it would be proper to separate lignin and fungus before ultrafiltration to keep the membrane clean.

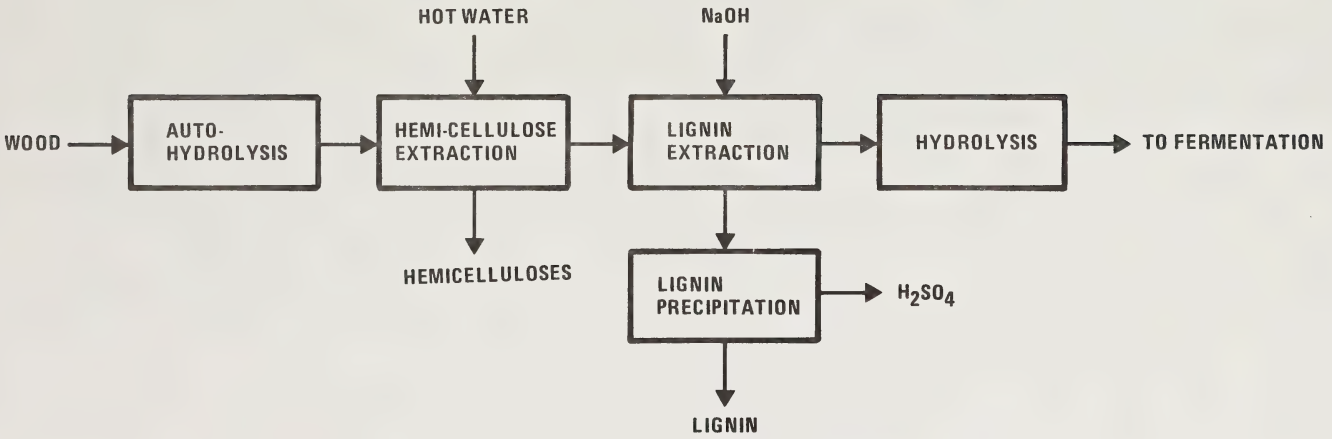
If an acid hydrolysis process is used, the lignin will be badly charred, and will probably be suitable only for use as fuel after careful removal of all sulphuric acid. When batch percolators are used for acid hydrolysis, as in the Scholler process and its variations, lignin accumulates at the bottom of the percolator and is removed at the end of each run. In the other acid hydrolysis processes discussed, lignin will be carried along with the sugar solution. It may be filtered ahead of the lime precipitation, washed and dried for use as fuel.

Hemicellulose Separation and Conversion

As shown in Figure B-9a, the hemicellulose fraction can be separated upstream of hydrolysis. However, in this case the hexose sugar component of the hemicelluloses is lost to fermentation. If not separated before hydrolysis, pentose sugars, which constitute the bulk of hemicelluloses, are retained in the stillage after ethanol is stripped off.

A bacterium, Clostridium acetobutylicum, converts both hexoses and pentoses to butanol, an alcohol which has many advantages over ethanol as fuel and for industrial purposes. Some acetone and ethanol is also produced by this bacterium. The fermentation butanol process was practiced for many years until cheaper petrochemical processes displaced the fermentation process. Much attention is now being paid to refurbishing and perhaps starting up the old butanol plants; also to modernizing and improving the process. There is no doubt that fermentation butanol will once again take its place in the market. Its ability to convert both classes of sugars present in poplar wood to an alcohol makes it particularly suitable in the present context and at some point, this process will need very careful study as an alternative approach to produce liquid fuels from poplar.

a) LIGNIN SEPARATION BEFORE HYDROLYSIS




b) LIGNIN SEPARATION AFTER HYDROLYSIS

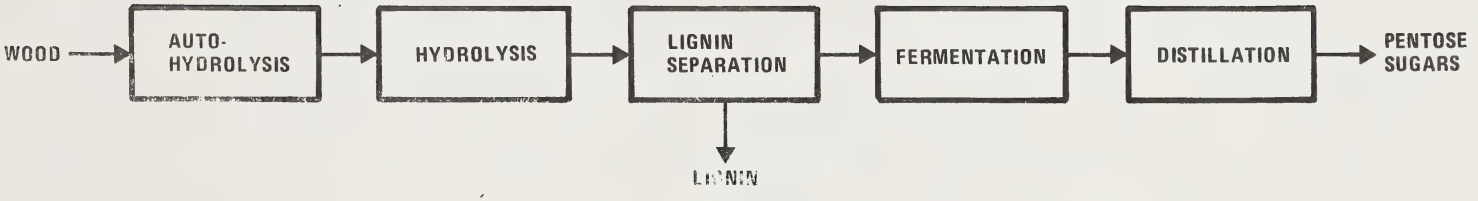



FIGURE B-9

The pentose sugars can also be used to grow Torula yeast or single cell protein. This practice has been used at Attisholtz plant in Switzerland. Single cell protein for human consumption is also produced at Boise Cascade Paper Company's Salem, Oregon, plant, using spent sulphite liquor, and at Monarch Paper Corp. at Rhinelander, Wisconsin.

## Conclusions

This review indicates rapid development in the technology of ethanol production from cellulose. Perhaps the most important development is the advent of autohydrolysis which has brought back into the picture processes not technically or economically feasible in the past. The emphasis now is on the use of autohydrolyzed wood to achieve high yields, high sugar concentrations and improved economics.

Rapid developments are taking place in enzyme production technology and are expected to continue. Potential exists to reduce the cost of enzyme production to that comparable with grain ethanol. This is expected to be achieved by increasing the enzyme activity using mutation and selection techniques. Another area, where rapid developments are expected, is pentose fermentation. However, practical application is foreseen if simultaneous fermentation of hexoses and pentoses can be performed.

The conventional processes for acid hydrolysis result in low yields and low sugar concentrations. However, new concepts are being developed and could result in high conversion efficiencies when combined with autohydrolysis.

Work continues on the development of fermentation, distillation and integrated fermentation-distillation to improve energy efficiency. Most of this work is based on conventional ethanol substrates and technology will be commercialized in sugar and grain alcohol industries initially. It is our opinion that these new processes need not be tested at the proposed plant to help focus on the key areas of pretreatment, hydrolysis and by-products.

APPENDIX D
PATENT SEARCH

MORRIS WAYMAN LIMITED
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Sept. 11, 1981

Dr. H. Husain,
W.L. Wardrop & Associates Ltd.,
77 Main Street,
Winnipeg, Manitoba
R3C 3H1

Dear Hadi,

Alberta Aspen Alcohol Facility
Patent Search

Enclosed Please find the first report of Mr. Robert Hirons, of Hirons, Rogers & Scott, Patent Agents, of Toronto. Mr. Hirons has had extensive experience in the field of wood autohydrolysis and related topics, having acted for me, for Stake Technology and for other players in the field. The search is limited to Canada, and to the last 30 years. Should technology be developed in Alberta for export, the patent situation in other countries can then be reviewed. The search left a few questions unanswered, and I shall give you a supplementary report shortly. Here is a summary of the situation as I see it now.

The most serious patent which may have to dealt with by the owners of the Alberta Aspen Alcohol Facility is that issued to Dietrichs, Sinner, Opderbeck and Brachthaüser, No. 1,087,122, October 7, 1980, a copy of which is enclosed. This covers the autohydrolysis step in the preparation of glucose in the range of $160^{\circ}$ - $230^{\circ}$C for 2 to 480 minutes, when coupled with alkaline extraction. Thus autohydrolysis of the Stake type is covered, but only when accompanied (followed) by lignin extraction. Neither the Iotech nor the Wenger types of autohydrolysis are covered by this patent.

I have recently been in touch with the parent German company, Dechema, of Frankfurt, copy enclosed, and Dr. Buchholz (beechwood) states that the focus of the autohydrolysis work has moved with Dr. Sinner. Dechema continues to work on enzyme hydrolysis. Also I have a recent letter from I.F.P. (Institut Francais du Pétrole) who have adopted autohydrolysis. I have asked them what equipment they use.

The same group own other patents, mainly dealing with the isolation of xylan and such. We need not be concerned. But at some point, Alberta should contact this group, although it is very difficult to know whom to contact since Dr. Dietrichs has died and the group itself has disintegrated. Still, someone owns the patents.

Hydrolysis of cellulosic material for sugar production, for fermentation or otherwise, is old, and a Canadian patent, No. 519021 of 1955, reviews the earlier practice. The patent itself is not of any consequence, since it is directed to the isolation of crystalline glucose.

Enclosed also for your information are two Stake patents issued January 1980, covering their equipment. If equipment is bought from Stake the terms of purchase would have to provide for protection against these patents.

On enzyme hydrolysis, No. 1,051,803 appears to cover the concept practiced at Gulf Kansas using the aqueous culture mass of the enzyme preparation process as the hydrolysing agent. I have requested a copy of this patent. There are no patents issued in the name of Iotech, nor relevant patents to Gulf Canada. We heard that a patent had issued to E.A. DeLong, formerly President of Iotech, and I have asked to have this located if it exists.

This covers the subject to the moment. If more develops, I shall let you know.

Yours very truly,

Morris Wayman

# *Hirons, Rogers & Scott*

PATENT AND TRADEMARK AGENTS

*From our Toronto Office*

*Stanley J. Rogers, B.Sc.*
*Robert G. Hirons, B.Sc.*
*Christopher R. Scott, B.Sc.*
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August 31, 1981

Prof. Morris Wayman
Morris Wayman Limited
Consultants
17 Noel Avenue
Toronto, Ontario
M4G 1B2

Dear Prof. Wayman:

Re: Alberta Aspen Alcohol Facility - Patent Search
Our File: 1058

In accordance with our discussion and your instruction, I have now conducted a "state of the art" search through Canadian patents, to locate and summarize patents of interest to you in connection with the above project.

In conducting the search, I have had in mind the Departmental "Terms of Reference" set out and attached to the February 27, 1981 letter from the Department, and your handwritten note to me . Thus I have looked at the four listed topics, namely:

1. Wood steaming at high temperatures for short times;

2. Acid hydrolysis of wood;

3. Enzyme hydrolysis of wood;

4. Alcohol from cellulose or wood

Some difficulties have been encountered in the search, arising out of the breadth of these definitions, and the iodosyncracies of the Canadian patent system.

One particular problem area for this search arose in connection with the acid hydrolysis of wood. This is found in Class 260-232, organic compounds, carbohydrates and derivatives, polysaccharides, acid hydrolysis of cellulosic materials. This

of course covers many old and well-known chemical pulping processes. The entire class is related to saccharification of cellulose, i.e. sugar production, using acid hydrolysis of the cellulose. The class definition sub-notes say that the class is especially for depolymerization of ligno-cellulosic material, e.g. sawdust with dilute acid boiling, and then acid saccharification. Sugars suitable for conversion to alcohol might well be the products of such processes. I have therefore limited this aspect, somewhat arbitrarily, to patents issued in the last 25 years, and report apparently relevant ones below. I have noted a number of earlier patents, and can review these again if you wish. It will be noted that this class includes one particularly relevant patent by Dietrichs.

Patents from Class 260-232 - Acid hydrolysis

1,100,492 Continue hydrolysis to produce sugars is effected by cyclically immersing a solid divided ligno cellulose material in a bath of concentrated hydrochloric acid and draining the material between successive immersions so as to dissolve the produced sugars, until the sugar concentration of the acid in the bath has attained a desired value.

1,087,122 Dietrichs - Process for the production of glucose from cellulose raw material which can be disintegrated by steam pressure treatment and defibrination, the raw materials being treated with saturated steam at 160-230°C for 2 minutes - 4 hours, the vegetable raw material disintegrated in this way is solvent extracted with aqueous alkali, and the fibrinous residue subjected to acid or enzymatic hydrolysis. (copy enclosed)

1,076,109 Continuous hydrolysis of a vegetable solid pentosan containing material in a temperature range 80-120°C and pH below 5 with extraction of a useable hydrolysis residue, by feed of raw material at one end and feedout at the other end of a hydrolysis vessel. Parts of the hydrolysis fluid are re-circulated into the hydrolysis vessel at two different concentrations at different or identical levels.

1,051,884 Continuous method for decomposition of cellulosic raw material by acid hydrolysis. The raw material is continuously hydrolyzed in one reactor but in two steps, first in the upper reactor in vapour phase and second in the bottom of the reactor in a liquid phase to decompose disaccharides and trisaccharides formed in the first stage.

1,010,859 A xylan containing raw material is first heated in water vapour and then impregnated with cold acid so as to cause the water vapour in the pores of the material to condense and create a negative pressure

in the pores. The material is then placed in a water vapour atmosphere to hydrolyze and the resultant xylose extracted with water.

924,713 Chemical degradation of cellulosic material is accomplished by percolating a liquid agent through the material in a closed percolation vessel having two superposed filter areas of downwardly diminishing cross-section. The material is supported at a level above the filter areas. The liquid reaction agent is introduced from below through the upper of the filter areas into the cellulosic material in an amount sufficient to cover the material. Steam is simultaneously introduced through the lower of the two filter areas to raise the temperature in the vessel to at least 100°C.

919,664 Carbohydrates are manufactured from xylan and cellulose containing raw materials by acid hydrolysis, separation of precipitated lignin, and passing the hydrolyzed aqueous medium through an ion exchange resin bed followed by extraction with methanol.

904,273 Xylose and xylitol are produced from cellulose materials with an acid agent followed by hydrogenation of the resulting mixture to form xylitol from xylose.

835,952 Sugars and other chemicals are produced by treating a cellulose containing material with steam in a closed chamber in the presence of acid under mild conditions, and withdrawing the resulting sugar-containing condensate from the chamber. The remaining material is then carbonized at higher temperatures to produce charcoal.

663,702 Cellulose-containing material is saccharified with hydrogen chloride in a gas suspension method, with addition of a dry substance, e.g. lignin with smaller average size than that of the cellulose-containing material.

618,551 Cellulosic lignin-containing materials are hydrolyzed by treatment at 15-30°C with hydrochloric acid solution in a substantially anhydrous aliphatic alcohol to obtain essentially pure cellulose and a solution of sugars and lignin, which is subsequently separated.

585,424 Cellulosic substances are saccharified with a first preliminary hydrolysis step with hydrochloric acid and a second saccharifying step with concentrated hydrochloric acid in the 40-41% concentration range. The acid concentration in the first step is 34-37% by weight.

573,376 Two step hydrochloric acid saccharification of cellulosic materials, using hydrochloric acid of 29-37% at 15-30°C for the first step, in an alphatic alcohol solvent, and hydrochloric acid of 39-42% concentration for the second step.

532,353 Wood is hydrolyzed with concentrated hydrochloric acid in a series of separate stationary columns, connected to form a saccharification unit, with circulation of water and hydrochloric acid from one column to the next.

✓519,021 Cellulosic materials rich in pentosan are processed by preliminary hydrolysis, followed by saccharification, recovery of grape sugar, and fermentation to produce alcohol (this patent has been purchased).

516,924 Crystalline glucose is produced from wood by treating the wood with dilute acid at 110-140°C, hydrolyzing the solid residue with concentrated hydrochloric acid, adjusting the sugar concentration and hydrochloric acid concentration in the residual mixture, heating for 1-2 hours to 110-140°C, and subjecting to ion exchange to remove the acid without introducing salts, and then concentration to crystalize the glucose.

508,915 Cellulosic materials are hydrolyzed by treatment in counter-current flow with dilute hydrolyzing solution and steam, under super atmospheric pressure, to produce sugar, lignin and volatile products, and the mixture so formed subsequently separated.

That is summary of all patents in this class believed to be relevant, back to those issued in 1952.

To locate patents relating to steam digestion of wood, I searched, after consulting classification examiner, class 92-57.3 which is part of the class relating to paper making and fibre liberation, specifically "paper-making, stock treatment, digestion, systems with steam supply, generation or recovery". The following patents were noted:

1,063,407 Method and apparatus for minimizing steam consumption in the production of pulp used for the manufacter of fibre board and the like, in which chips of cellulose material are ground in a defibrator or refiner in an environment of saturated steam above 100°C and corresponding steam pressures. The chips are normally pre-steamed with atmospheric steam, separated from the pulp at the discharge end of the defibrating process to a temperature between 90 and 100°C and compressed and de-watered to a dryness of at least 50% and then passed into a pre-heater.

991,462 Method of obtaining homogeneous digestion in the periodic cooking of ligno cellulose material utilizing at least 1 surge-boil stage carried out by pressure reduction wherein steam is supplied directly to the digestor bottom simultaneously with the pressure reduction. This has the effect of rendering the system less sensitive to over-pressure at the surge-boil stage.

I also searched in certain sub-classes of class 9, "paper making chemistry", specifically sub-classes 10 - digestion with inorganic acids, 11 - digestion with nitric or nitrous acids, 12 - digestion with sulfur acids, and 70 - miscellaneous wood treatments.

All the patents in these patents relating to wood treatment concerned the manufacture of pulp material, not chemical breakdown of the cellulose into saccharides or the like and so no relevant patents were noted in this class 9.

To find patents relating to enzyme hydrolysis of wood, and fermentation of cellulose-derived sugars to alcohol, etc.

Class 195, microbiochemistry, was searched. In particular, sub-class 2, the generic class for processes using, producing or compositions containing a microbe or enzyme was searched. As used in the Patent Office classification, an enzyme is a chemical produced by an organism which acts as a catalyst for chemical reaction. This general class turns out to have large numbers of patents relating to beverages, spirit production and the like, which have been ignored.

Sub-class 1 was inspected. Of possible interest in this sub-class is Canadian Patent 578,513, relating to fermentation of pentosan-rich solutions.

Sub-class 129, processes, specific product production, alcohols and related by-products, was recommended by the examiner as relevant. This sub-class contains large numbers of beverage patents, but the following may be of interest:

200,690 Methyl and ethyl alcohol produced from waste sulphite cellulose lye.

945,090 Manufacture of carbohydrates from lignin-free hemi-celluloses, wherein polysaccharides have been converted to monosaccharides by subjecting an aqueous medium containing said materials to fermentation to yield a solution containing xylose free of hexoses and other pentoses, and thereafter, if desired, hydrogenating the aqueous medium to yield xylitol.

Sub-class 130 was searched -- this has the same

heading as 129, except that the specific products produced are butyl alcohol, acetone, butyric acid. The following may be of interest:

1,093,487 One step production of acetone and butanol from cellulose using a cellulase and an acetone/butanol producing microorganism in simultaneous reaction on the cellulose.

Sub-class 139 was searched, relating to microbiochemical processes, specific products production, saccharides.

763,408 describes a process of subjecting B-glucans to the cellulolytic fermentation of a fungus of the genus Oxyporus. The process is applied to cellulose powder, filter paper, cotton, etc., but there is no specific disclosure of its use on wood.

1,051,803 relates to enzymatic hydrolysis of cellulose to obtain water soluble sugars, in which the enzyme source is the aqueous culture mass of the enzyme preparation process.

(1,087,122 Dietrichs also shows up in this class.)

Sub-class 140 of Class 195 was searched, wherein the specific product produced is polysaccharides (dextran), but nothing was found relevant to wood.

Class 260-641.8 was searched, again on an examiner's recommendation, to find other patents relating to alcohol from wood. The heading for this sub-class is "organic compounds, alcohols, treatment purification or recovery, aliphatic alcohols, acyclic saturated monoalcohols, methanol and ethanol". This patent classification contains some very old patents relating to the distillation of wood, to produce wood alcohol (methanol), including the following:

109,313 Manufacture of wood alcohol by distillation and progressive reduction of the temperature of crude pyrolignious acid vapours from the retort.

142,269 and 146,254 Production of methyl alcohol from wood by boiling with water and acid or alkali and collecting and condensing the vapours formed.

402,583 Preparation of methanol by catalytic hydrogenation of lignin, over a catalyst such as nickel.

The class also contains several patents relating to the manufacture of alcohol from sulphite cellulose liquors, namely the following:

188,638
202,945
405,631
1,088,957

Finally, I conducted name searches under the suggested names.

A name search under Stake Technology Ltd. reveals the following issued Canadian patents (others must be pending):

1,070,537
1,070,646

Both of these are enclosed. The basic process case of Bender appears not yet to have issued in Canada, but we can supply a copy of the issued U.S. equivalent.

No patents have issued in the name of Iotech in the period 1967 to date.

An assignee search 1972 to date under Gulf Canada, Gulf Oil and Gulf Research & Development reveals no patents apparently relevant from their titles to wood treatment and extraction processes.

A search in the name of Hans-Hermann Dietrichs revealed three recently issued Canadian patents, of which you were previously aware of two, I believe. A copy of each of the three is enclosed.

Please let me know if we can go further into this matter, or obtain copies of any additional patents referred to above. Our account for services and disbursements is enclosed.

Yours very truly

HIRONS, ROGERS & SCOTT

Robert G. Hirons

ROBERT G. HIRONS

RGH:sc
Encls.

Dr. K. Buchholz c/ DECHEMA Deutsche Gesellschaft für chemisches Apparatewesen e.V.

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Kernzeit Montag–Freitag
von 9.30–16.00 Uhr

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Dr. M. Wayman
Morris Wayman Ltd.
Consultants
17 Noel Avenue

CDN-Toronto M4G 182

Ihr Zeichen
Ihr Schreiben vom
Unser Zeichen DI/KBz/uf
Tag August 28, 1981

Betrifft

DECHEMA

Hausruf (Durchwahl)
0611/7564 -370

Dear Dr. Wayman,

I apologise for my late answer to your letter of July 6.

Equipment for high temperature steaming is not on the market, as far as I know. A pilot plant has been developed by Dr. Puls and Dr. Sinner, who now continues the development at

Fritz Werner Industrie-Ausrüstungen GmbH
P.O. Box 12 54
D-6222 Geisenheim .

As to tubular enzyme reactors, we developed a continuous tubular reactor with a screw for transportation of cellulosic matter on a laboratory scale. Experiments are running, and a scale-up is envisaged for 1982. We are not concerned with acid hydrolysis.

I hope that this information is helpful.

Sincerely yours,

K. Buchholz

APPENDIX E

**PROCESS AND EQUIPMENT TESTS**

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November 6, 1981

Dr. H. Husain,
W.L. Wardrop & Associates,
77 Main Street,
Winnipeg, Manitoba
R3C 3H1

Dear Hadi,

Wenger Pulps from Alberta Aspen

In early September, four samples of Alberta aspen were sent to Wenger Manufacturing, of Sabetha, Kansas, for extruder processing. Each of these was processed in Wenger's pilot plant, the conditions being somewhat different for each wood sample. Portions of the resulting pulps were sent to my laboratory at the University of Toronto for enzyme hydrolysis. The results of these tests were generally satisfactory, and are given here.

1. Processing Conditions

As reported by Wenger in their letter of September 14, processing conditions were as follows:

| Aspen sample | Temperature | Pressure | Feed Rate: kg/h |
|---|---|---|---|
| A | 246°C | 1200 psi | 60 |
| B | 246 | 1400 | 60 |
| C | 249 | 2000 | 136 |
| 80%A+20%C | 260 | 1500 | 60 |

The variation in temperature and pressure are probably largely planned, but leave open the question of the controllability of the Wenger extruder. Sample C was not well disintegrated, which suggests that it may have been passed through the machine too rapidly. The other three samples were very well defiberized, and would readily be handled in conventional processing equipment.

2. Yield

The run of mixed A+C was carried out more or less quantitatively. Of 45.4 kg moist chips = 22.5 kg dry basis, 18.4 kg dry fibre were recovered after the extruder, a yield of 81.8%. This represents a loss of 18.2% of the wood substance, which is considerably higher than the volatiles loss of 12.3%

found when treating aspen wood in the Stake autohydrolyser, as reported in the paper by Wayman, Lora and Gulbinas (A.C.S. Symposium Series No. 90, p. 193). This is somewhat worrisome, and suggests the need for careful study of processing conditions in the Wenger extruder in the demonstration alcohol plant.

3. Enzyme Hydrolysis

These samples were hydrolysed with a mixture of cellulase (Novo), cellobiose (Novo) and hemicellulase (Miles). Two levels of enzyme were employed, 10% and 20%, and two times, 24 h and 48 h, at 50°C, pH 4.8. Hydrolysis was followed by glucose production, with these results:

| Aspen sample | alpha cellulose | Glucose Yields Based on Alpha Cellulose 10% enzyme 24h | 10% enzyme 48h | 20% enzyme 24h | 20% enzyme 48h |
|---|---|---|---|---|---|
| A | 48.4% | 70.5% | 83.2% | 77.2% | 95.4% |
| B | 45.0 | 50.9 | 67.7 | 70.9 | 98.8 |
| C | 47.0 | 49.9 | 62.3 | 74.5 | 91.9 |
| A+C | 44.0 | 61.6 | 76.0 | 96.3 | 104.1 |

Satisfactory 90+% hydrolysis was obtained in all cases with 20% enzyme acting for 48 hours. The discrepancy in the A+C yield of over 100% is attributed to a probable low alpha cellulose determination, the calculated alpha from the A and C values being 47.5%. If this is used in the calculation, the glucose yields come into line with the other results (89.2 and 96.4%, respectively).

4. Conclusions

As a pretreatment for aspen wood, the Wenger extruder is quite satisfactory in preparing the wood physically for further processing, and in sensitizing it for enzyme hydrolysis. In the one quantitative run, the loss during extrusion was significantly greater than expected, which should be studied in the demonstration alcohol plant.

Cordially,

Morris Wayman

Morris Wayman

WENGER MANUFACTURING
INDUSTRIAL FOOD AND GRAIN PROCESSING MACHINERY
EXTRUSION COOKING SYSTEMS

PLANT & GENERAL OFFICE
SABETHA, KANSAS 66534

PHONE 913 / 284-2133
CABLE: WENGERMIX

September 14, 1981

W. L. WARDROP & ASSOCIATES, LTD.
Engineering Consultants
H. (Hadi) Husain, Phd., P. Eng.
77 Main Street
Winapeg, Manatoba Canada R3C 3H1

Mr. H. Husain,

In response to your telex regarding tests on Alberta Hardwoods, we have completed four extruder runs. Data from these runs is as follows:

| | RUN #1 | RUN #2 | RUN #3 | RUN #4 |
|---|---|---|---|---|
| Woodchip Type | A | B | C | 80% A & 20% C |
| Extruder Pressure | 1200 PSI | 1400 PSI | 2000 PSI | 1500 PSI |
| Extruder Temp. | 475°F | 475°F | 480°F | 500°F |
| Feed Rate | 130lbs/hr | 130lbs/hr | 300lbs/hr | 130lbs/hr |

During Run #4, 100 lbs. of woodchips were weighed out and put into the extruder. Of this 100 lbs. chips @ 50.5% MCWB, 64.2 lbs. @ 36.8% MCWB were recovered. Thus a yield of 82% was achieved. This test should be duplicated for reliability.

Other analysis being done on these samples are (1) enzyme solubilities and (2) cellulose content or available sugar content. We will send these results and information pertaining to condensate upon completion of testing.

If you have any questions concerning this data or testing procedures, I will be happy to help you.

Sincerely,

Gordon R. Huber
Director of Biomas
Utilization

CC: Dr. Wayman, Terry Rachuk, Gary Edelman
GRH/krk

81-7028-01-00.

+
WARDROP WPG

R AND M REED
11-2-81
ATT: DR HUSAIN
CONFIRMING PHONE CALL: WOOD CHIP SLURRY AT 30 PERCENT SOLIDS BY
WEIGHT PUMPED SATISFACTORILY WITH OPEN THROAT MOYNO PUMP FRAME 2J6
USING A BUNA N SYNTHETIC RUBBER STATOR
IN FACT SOLIDS WERE INCREASED TO 40 PERCENT AND SLIPPAGE IN PUMP WAS
ONLY 10 PERCENT
PUMP SPEED WAS 107 RPM
WE CAN SHIP WOOD CHIPS WHEN YOU HAVE SHIPPING ADDRESS
REGARDS
W H WOODS
R AND M REED

APPENDIX F

**RESEARCH AND DEVELOPMENT REQUIREMENTS**

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## Introduction and Crucial Problems

It is widely accepted that cellulosics are destined to make a major contribution to liquid fuel supply, yet there is as yet no functioning cellulosic alcohol plant in the western world. There is no agreement on a preferred process and doubt even yet about the economic viability of the proposed process. There are a number of key questions related to the conversion of cellulosics to alcohol which still require research answers. Some of these are engineering questions, some are chemical and perhaps the most serious are microbiological. To highlight briefly these main questions will help understand the nature of the research and development program. The crucial problems may be summarized as follows:

### Microbiological

- the production of cheap, effective cellulase from various fungi or other sources.

- identification and development of pentose-fermenting micro-organisms.

- selection of best alcohol yeast or bacterial species and strains, their adaption to wood substrate, mutagenesis for improved strains and genetic engineering for specific purposes.
- immobilized yeast cells for rapid, efficient fermentation.

Engineering Problems

- percolator design, including materials of construction, for acid hydrolysis.

- achieving and maintaining high sugar concentrations in saccharification.

- sugar separation from undissolved fibre, lignin and micro-organisms in maximum yield and minimum dilution.

- enzyme saccharification tanks with minimum energy requirement for agitation.

Chemical Problems

- pretreatment with minimum formation of furfural and other substances toxic to micro-organisms.

- sugar solution neutralization techniques to remove toxic substances.

- separation and purification of lignin as an article of commence, within the process and its conversion to useful chemicals.

It is apparent that the research and development program will have strong microbiological, engineering and chemical components as well as other scientific capability.

Some of the questions asked need to be studied on the very small scale, others can be studied in the pilot plant. For many of these questions, facilities intermediate in size between laboratory and pilot plant, that is bench scale equipment, will be required. The laboratory design will address both scales: "laboratory" and "bench scale".

## Microbiological Research Program

### Cellulase Production

The aim here is to produce cheap, effective cellulase, which will be of acceptable cost and act rapidly so as to reduce capital costs. Since enzyme hydrolysis of grain starches (corn in particular) has been widely practised commercially for many years, this is not an unrealistic expectation. It is unlikely that such cellulase can be produced by simple improvement of Trichoderma viride. The enzyme system has several components and the products of two or three fungi or bacteria may be required. A broad innovative attack on this key problem is required.

### Pentose Fermentation

This is an important problem nearing solution. Two approaches are well advanced:

i) the use of isomerase enzyme for converting pentoses to fermentable sugars, and

ii) the identification of yeast, bacteria and fungi able to ferment pentoses.

Of these, the second seems to be well ahead. This area needs detailed attention.

## Alcoholic Fermentation

While ordinary yeasts, such as *Saccharomyces cerevisiae* and others are reasonably satisfactory, any micro-organism can be much improved in efficiency by selection, by adaptation to substrate (in this case aspen or agricultural residues), by mutagenesis and further selection, and by genetic engineering for specific improvement.

Several studies recently suggest that a bacterium *Zymomonas mobilis* may be advantageous in alcoholic fermentation. The MIT group has suggested bacteria which are capable of both saccharification and fermentation, while the Penn State group has developed a bacterium capable of quantitative conversion of pentoses to alcohol. These findings require investigation.

## Immobilized Cells

Recent experience suggests that very high inocula and immobilized yeast cells are efficient, rapid converters of sugars to alcohol. Total fermentation time of nine and one-half hours being reported. This should also be in the program.

## Simultaneous Saccharification-Fermentation

While this technique is not as much discussed today as it was a few years ago, it may well be adapted to enzyme hydrolysis as a means of shortening the total time from cellulose to alcohol.

## Solid State Fermentation

This method has been used with success on agricultural residues and should be studied here.

## Chemical Research Program

### Pretreatments

The chemistry of pretreatment needs to be better understood to achieve maximum benefit in terms of ease of alcohol production, minimum destruction of hemicellulose with attendant formation of toxic by-products, maximum ease of lignin separation with minimal attack on lignin structure. The pretreatments to be studied include the steam-explosion, cooker-extruder and mechanical roll-milling.

In the steam-explosion and cooker-extruder pretreatments, the effects of catalysts such as sulphurous acid and others need study.

### Enzyme Hydrolysis

This is a question of having the proper enzyme system formulation, including the different enzyme components, catalysts such as Vitamin $B_{12}$ and inorganic trace elements. Studies of various enzyme sources, alone and in combination, and optional conditions of time, temperature, pH and consistency are required. The fed-batch method of hydrolysis should be studied.

Partial hydrolysis with recycle and multi-stage enzyme hydrolysis are promising avenues for improvement. These recognize a fundamental fact of cellulose structure, that any cellulose contains readily hydrolysable cellulose and difficultly hydrolysable cellulose. While this is most apparent in acid hydrolysis, it applies also in enzyme hydrolysis.

## Enzyme Recovery and Recycle

At the end of saccharification, the sugar solution contains enzyme which should be recoverable by simple ultrafiltration for recycle. It is also suggested that contacting the sugar solution with incoming feed fibre will result in absorption of enzyme by the fibre. While there are obvious mechanical problems here, these methods merit study to reduce enzyme cost.

## Acid Hydrolysis

The basic kinetics of this process are known for pure cellulose but not for poplar wood. Previous work has taken little or no account of the inhomogeneous structure of wood cellulose, particularly the presence of readily hydrolysable and difficultly hydrolysable fractions. Hence, the two-stage and multi-stage processes proposed for the pilot plant. The kinetics of poplar acid hydrolysis needs re-examination taking this inhomogeneity into account, in order to define optimal operating conditions and optimize sugar yields. For these studies the hydrolysis process should be seen as a succession of autohydrolysis, lower temperature acid hydrolysis and higher temperature acid hydrolysis, with sulphuric acid concentration as another variable. Partial hydrolysis with recycle requires study also.

This process can be varied in another important respect by removal of lignin and hemicellulose following autohydrolysis before proceeding to the multi-stage acid hydrolysis.

## Lignin Separation, Purification and Conversion

Following autohydrolysis, lignin becomes soluble in alkaline solutions from which it may be recovered by lowering the pH. It may be purified by resolution in alkali and reprecipitation. It may then be spray dried. This is a very pure lignin suitable as a therapeutic dietary additive for medical research (see special section on lignin marketing) or it may be converted to aldehydes or other useful chemicals. These processes require research.

## Hemicellulose Separation

In the lignin recovery step of 3.5, the filtrate from the lignin contains the hemicellulose. This may be fermented, or it may be used to grow single cell protein, or it may be evaporated to a wood molasses suitable for cattle feed. These processes and their relative economics require investigation.

## Volatiles

The pretreatment step produces acetic acid, methanol, furfural and other volatiles. These need research to establish their value, and how their value is affected by pretreatment operating conditions.

## Organosolv Pulping

Several research groups (Kleinert, Pye, April, Paszner) have suggested cellulose purification using organic solvent ahead of hydrolysis. This is very attractive when the solvent is ethanol, or a mixture of ethanol and a catalyst to saccharify wood carbohydrates. This needs to be in the research program as an alternative approach to pretreatment.

## Distillation

The chemistry of the alcohol produced in the process should be determined, especially the content of alcohols other than ethanol, and other organics.

## Stillage

The usefulness and marketability of the stillage in various forms may depend on the process scheme. Under certain conditions, it may contribute significantly to income. The environmental impact of other disposal methods needs to be considered.

## Dehydration

The system for producing anhydrous alcohol should be under continual study. In addition to azeotropic distillation methods, membrane separation and solid dehydration have been proposed.

## Agricultural Crop Residues

The research program should include processes for conversion to alcohol of straw, oilseed stalks, sugarbeet pulp, and other agricultural crop and food industry residues which have the potential of contributing significantly to alcohol supply.

## Cold Strong Acid Hydrolysis

Research on cold strong acid wood hydrolysis, especially that employing 41+% hydrochloric acid is of interest because of its presumed capital cost advantages.

## Engineering Research Program

### Reactor Design

There is a great need for design of reactors for acid hydrolysis. The special areas requiring research are materials of construction, percolators with continuous drainage systems, valving, feed and discharge systems for continuous and batch reactors.

### Solid-Liquid Separation

The objective here is the development of equipment for achieving maximum sugar solution recovery from a mixture including fibre, lignin and other solids. The types of equipment available include drum filters, presses, centrifuges, belt filters all equipped with spray washers, some with counter current flow. This type of separation is needed in saccharification, and particularly in lignin recovery.

### Agitation of High Consistency Pulp

In enzyme saccharification, a modest amount of agitation is required. Since one objective is to maintain very high sugar concentrations, the fibre ratio must be high and then agitation becomes a problem. This is helped by fed-batch operation, but the basic problem remains. It needs attention.

### Low-Energy Fermentation-Distillation Systems

A number of proposals have been offered for low-energy fermentation-distillation systems. These are not in commercial operation, although a few rather substantial pilot plants are in operation, (Alfa-Laval, Dechema). Some systems are based on heat exchange, others on vacuum fermentation, still others combine both concepts. These designs and their evaluation, as well as their economics, require constant investigation.

## Lignin Separation

Lignin produced during wood hydrolisis is in the form of very fine particles, difficult to separate from solution. Means of improving this separation are required.

## Product Marketing and Economic Research

### Alcohol

The performance of the ethanol produced in this plant in internal combustion engines, spark and diesel, automotive and stationary, needs to be adequately monitored. This should be done at various proof levels, up to the anhydrous and with various contaminants and additives.

The purification of alcohol for industrial use, which commands a higher price and its conversion to other chemicals and useful products should also be investigated.

### Volatiles

The volatiles from pretreatment include furfural and a number of other valuable chemicals. The potential products, conversion products and markets need study. This may be particularly true of furfural from agricultural residues.

### Lignin

This chemical constitutes about 17 - 20% of the poplar wood, and its upgrading can represent substantial additional income for the project. Lignin itself can command several markets, including dietary and medical and fibreboard and particleboard adhesives. It may be converted to surface active agents for enhanced oil recovery and oil well drilling mud viscosity control, where it is well established. It can also be converted to chemicals such as vanillin and syringaldehyde, which have good markets and the compounds described by Professor J.M. Pepper of the University of Saskatchewan. A lignin plant as part of an alcohol complex could markedly improve the overall plant economics.

## Hemicellulose

The hemicellulose can be concentrated to form wood molasses, or fermented to single cell protein. It is also a source of xylose, which may be reduced to xylitol, a sweetener, or converted to furfural. The economics of these various processes need to be monitored.

## Other Fermentation Products

The end product of saccharification is a sugar solution, which may be fermented to ethanol. There are however, a great many other potential uses for this sugar solution, especially as a source of fermentation products. These include butanol, acetone, penicillin and other antibiotics, enzymes, baker's yeast, single cell protein, vitamins, and many others. These alternative possibilities should be under constant study. While ethanol may be the origin of this project, it is not necessarily the best way to make use of Alberta's poplar resource.

APPENDIX G
**CAPITAL COST ESTIMATES**

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

SHEET

X PY
GOING
FINAL

PROJECT ALBERTA POPLAR CHEMICAL PROCESS DEVELOPMENT & DEMONSTRATION FACILITY
EST. BY CH
PROJECT NO. 827028-01-00
COST/C.F.
COST/
SHEET 1 OF 2
LOCATION
COST/S.F.
DATE 19
OWNER
JOB NO.(OWNER)
DATE DUE 19

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | Installation & Indirect | | | Equipment | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Surge Bin | | | | | 1,100 | | 1,000 | 2,100. |
| | | Feeder | | | | | 58,300 | | 200,000 | 258,300. |
| | | Digester | | | | | 27,600 | | 30,000 | 57,600. |
| | | Flash Tank 1 | | | | | 5,700 | | 2,000 | 7,700. |
| | | Cooking Liquor Heat Exchanger | | | | | 70,000 | | 39,000 | 109,000. |
| | | Flash Tank 2 | | | | | 5,700 | | 2,000 | 7,700. |
| | | Blow Tank | | | | | 5,700 | | 2,000 | 7,700. |
| | | Cooking Liquor Heater | | | | | 26,700 | | 15,000 | 41,700. |
| | | Solvent Recovery Column | | | | | 97,900 | | 55,000 | 152,900. |
| | | Distillate - acetone condenser | | | | | 107,000 | | 60,000 | 167,000. |
| | | Distillage Surge Tank | | | | | 7,000 | | 4,000 | 11,000. |
| | | Distillation Column Reboiler | | | | | 53,500 | | 30,000 | 83,500. |
| | | Sugar Solution Holding Tanks | | | | | 17,400 | | 10,000 | 27,400. |
| | | Cooking Liquor Holding Tank | | | | | 48,700 | | 28,000 | 76,700. |
| | | Solvent Storage Tank | | | | | 3,500 | | 2,000 | 5,500. |
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W.L.WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

ESTIMATE SHEET
[X] WORKING COPY
[ ] BUDGET
[ ] FINAL

PROJECT ALBERTA POPLAR CHEMICAL PROCESS DEVELOPMENT & DEMONSTRATION FACILITY
EST. BY CH
PROJECT NO. 827028-01-00
COST/C.F. ____
COST/ ____
SHEET 2 OF 2
LOCATION ____
COST/S.F. ____
DATE ____ 19__
OWNER ____
JOB NO.(OWNER) ____
DATE DUE ____ 19__

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | Installation & Indirect | | | Equipment | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Acid Storage Tank | | | | | 3,500 | | 2,000 | 5,500. |
| | | Pretreatment Tank | | | | | 3,500 | | 2,000 | 5,500. |
| | | Pumps | | | | | 72,400 | | 30,000 | 102,400. |
| | | Mixers | | | | | 36,200 | | 15,000 | 51,200. |
| | | | | | | | | | | |
| | | Scrubber System | | | | | 35,700 | | 20,000 | 55,700. |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | TOTAL | | | | | 687,100. | | 549,000. | 1,236,100. |
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WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Site Work and Service Buildings
EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)
PROJECT NO. 817028-01
COST/
SHEET 1 OF 14
DATE 19
DATE DUE 19
LOCATION
OWNER

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Site Work, excluding Electrical | | | | | 130,000 | | 150,000 | 280,000 |
| | | Land | | | | | | | | 20,000 |
| | | | | | | | | | | |
| | | Building Costs | | | | | | | | |
| | | Pilot Plant Building | | | | | | | | |
| | | Piles & Foundation | | | | | 86,000 | | 120,000 | 206,000 |
| | | Structural | | | | | 430,000 | | 1,172,000 | 1,602,000 |
| | | HVAC | | | | | 50,000 | | 50,000 | 100,000 |
| | | Plumbing | | | | | 50,000 | | 50,000 | 100,000 |
| | | Electrical | | | | | 196,000 | | 200,000 | 396,000 |
| | | Covered Walkway | | | | | | | | 15,000 |
| | | Lab & Office Building | | | | | | | | |
| | | Piles & Foundation | | | | | 28,000 | | 30,000 | 58,000 |
| | | Structural | | | | | 125,000 | | 252,000 | 377,000 |
| | | HVAC | | | | | 33,000 | | 33,000 | 66,000 |
| | | Plumbing | | | | | 18,000 | | 18,000 | 36,000 |
| | | Electrical | | | | | 20,000 | | 24,000 | 44,000 |
| | | | | | | | | | | |
| | | | | | | | | | | 3,000,000 |

CONTINUED ON SHEET

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems Summary EST. BY _____ PROJECT NO. _____

_____ COST/C.F. _____ COST/ _____ SHEET 2 OF 14

LOCATION _____ COST/S.F. _____ DATE _____ 19____

OWNER _____ JOB NO.(OWNER) _____ DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| 0100 | | Pretreatment | | | | | 1,295,000 | | 1,775,000 | 3,070,000 |
| 0200 | | Enzyme Production & Hydrolysis | | | | | 1,541,000 | | 683,000 | 2,224,000 |
| 0300 | | Lignin Separation | | | | | 225,000 | | 310,000 | 535,000 |
| 0400 | | Fermentation | | | | | 670,000 | | 393,000 | 1,062,000 |
| 0500 | | Distillation | | | | | 604,000 | | 337,000 | 940,000 |
| 0600 | | Acid Hydrolysis | | | | | 424,000 | | 270,000 | 694,000 |
| 0700 | | Hemicellulose & Lignin Extraction | | | | | 1,078,030 | | 942,000 | 2,020,000 |
| 0800 | | Yeast Production | | | | | 311,000 | | 255,000 | 566,000 |
| | | | | Total | | | 6,148,000 | | 4,964,000 | 11,112,000 |
| CONTINUED ON SHEET: | | | | | | | | | | |

WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems
0100 Pretreatment
LOCATION
OWNER

EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)

PROJECT NO.
COST/
DATE 19
DATE DUE 19



| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Truck dumper | | | | | 31,370 | | 88,000 | 119,370 |
| | | Chip receiving bin | | | | | 5,720 | | 35,000 | 40,720 |
| | | Feeder | | | | | 8,740 | | 15,000 | 23,740 |
| | | Truck unloading conveyor | | | | | 107,170 | | 107,000 | 214,170 |
| | | Magnetic separator | | | | | 15,840 | | 31,000 | 46,840 |
| | | Discharge chutes | | | | | 12,160 | | 14,000 | 26,160 |
| | | Auxiliary chip conveyors | | | | | 62,990 | | 37,000 | 99,990 |
| | | Chip storage tanks | | | | | 96,690 | | 162,000 | 258,690 |
| | | Reclaim conveyors | | | | | 39,200 | | 26,000 | 65,200 |
| | | Main chip conveyor | | | | | 63,500 | | 30,000 | 93,500 |
| | | Chip screens | | | | | 26,470 | | 20,000 | 46,470 |
| | | Rechipper | | | | | 54,610 | | 40,000 | 94,610 |
| | | Distributing conveyor | | | | | 48,310 | | 36,000 | 84,310 |
| | | Receiving bins | | | | | 38,320 | | 60,000 | 98,320 |
| | | Iotech Reactor | | | | | 117,740 | | 75,000 | 192,740 |
| | | Wenger Extruder | | | | | 111,920 | | 314,000 | 425,920 |
| | | Stake System | | | | | 146,520 | | 154,000 | 300,520 |
| | | Surge tank and live bottom | | | | | 79,620 | | 148,000 | 227,620 |
| | | | | | | | | | | |
| CONTINUED ON SHEET: | | | | | | | | | | |

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems — EST. BY _____ — PROJECT NO. _____
0100 Pretreatment — COST/C.F. _____ — COST/ _____ — SHEET 4 OF 14
LOCATION _____ — COST/S.F. _____ — DATE _____ 19__
OWNER _____ — JOB NO.(OWNER) _____ — DATE DUE _____ 19__

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Scrubber system | | | | | 41,000 | | 23,000 | 64,000 |
| | | Controls | | | | | 187,250 | | 360,000 | 547,250 |
| | | | | | | | | | | |
| | | Total: | | | | | 1,295,140 | | 1,775,000 | 3,070,140 |
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WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems — EST. BY _____ — PROJECT NO. _____

0200 Enzyme Production & Hydrolysis — COST/C.F. _____ — COST/ _____ — SHEET 5 OF 14

LOCATION _____ — COST/S.F. _____ — DATE _____ 19____

OWNER _____ — JOB NO.(OWNER) _____ — DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Seed tank | | 2 | | | 4,570 | | 1,620 | 6,190 |
| | | Prefermenter | | 2 | | | 45,840 | | 16,160 | 62,000 |
| | | Production tank | | 6 | | | 1,179,780 | | 484,800 | 1,664,580 |
| | | Storage tank | | 1 | | | 172,300 | | 99,000 | 271,300 |
| | | Pulping tank | | | | | 6,240 | | 2,200 | 8,440 |
| | | Screw conveyor | | | | | 25,320 | | 15,000 | 40,320 |
| | | Screw conveyor | | | | | 5,630 | | 3,100 | 8,730 |
| | | Pumps | | 7 | | | 50,680 | | 21,000 | 71,680 |
| | | Screens | | 2 | | | 39,900 | | 20,000 | 59,900 |
| | | Air filter | | 1 | | | 11,230 | | 20,000 | 31,230 |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | Total: | | | | | 1,541,490 | | 682,880 | 2,224,370 |
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W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems — EST. BY _____ — PROJECT NO. _____
0300 Lignin Separation — COST/C.F. _____ — COST/ _____ — SHEET 6 OF 14
LOCATION _____ — COST/S.F. _____ — DATE _____ 19____
OWNER _____ — JOB NO.(OWNER) _____ — DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Sugar solution tank | | | | | 5,740 | | 3,300 | 9,040 |
| | | Repulp tank | | | | | 9,370 | | 3,300 | 12,670 |
| | | Washwater tank | | | | | 5,740 | | 3,300 | 9,040 |
| | | Low grade lignin tank | | | | | 17,210 | | 11,000 | 28,210 |
| | | Screw conveyor | | | | | 2,200 | | 1,300 | 3,500 |
| | | Screw conveyor | | | | | 2,200 | | 1,300 | 3,500 |
| | | Pumps | | | | | 28,960 | | 12,000 | 40,960 |
| | | Centrifuge | | | | | 153,870 | | 274,000 | 427,870 |
| | | | | | | | | | | |
| | | Total | | | | | 225,290 | | 309,500 | 534,790 |
| | | | | | | | | | | |
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WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems
0400 Fermentation
LOCATION
OWNER

EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)

PROJECT NO.
COST/
DATE
DATE DUE



| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Fermenters | | | | | 424,680 | | 149,600 | 574,280 |
| | | Beer well | | | | | 49,780 | | 28,600 | 78,380 |
| | | Pumps | | | | | 21,720 | | 9,000 | 30,720 |
| | | Air filter | | | | | 2,810 | | 5,000 | 7,810 |
| | | Centrifuge | | | | | 84,870 | | 150,000 | 234,870 |
| | | Cyclone | | | | | 780 | | 500 | 1,280 |
| | | Fan (blower) | | | | | 85,040 | | 50,000 | 135,040 |
| | | | | | | | | | | |
| | | Total: | | | | | 669,680 | | 392,700 | 1,062,380 |
| | | | | | | | | | | |
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| CONTINUED ON SHEET: | | | | | | | | | | |

WLW-4 77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems
0500 Distillation
LOCATION
OWNER

EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)

PROJECT NO.
COST/
DATE
DATE DUE

SHEET 8 OF 14
19
19

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | T. A. Tank | | | | | 1,420 | | 500 | 1,920 |
| | | F. O. Tank | | | | | 1,420 | | 500 | 1,920 |
| | | Decanter | | | | | 1,420 | | 500 | 1,920 |
| | | Dehyd. Agent Tank | | | | | 1,420 | | 500 | 1,920 |
| | | Product Storage Tank, Dewatering | | | | | | | | |
| | | & Dispensing Loadout | | | | | 57,920 | | 50,600 | 108,520 |
| | | Pumps | | | | | 57,920 | | 24,000 | 81,920 |
| | | Distillation Columns, Scrubbers, etc. | | | | | 356,520 | | 200,000 | 556,520 |
| | | Heat Exchangers | | | | | 89,130 | | 50,000 | 139,130 |
| | | Intermediate Product Tanks | | | | | 36,380 | | 10,000 | 46,380 |
| | | | | | | | | | | |
| | | Total: | | | | | 603,550 | | 336,600 | 940,150 |
| | | | | | | | | | | |
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WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems
0600 Acid Hydrolysis
LOCATION
OWNER

EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)

PROJECT NO.
COST/
DATE 19
DATE DUE 19



| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | LABOUR | | | MATERIAL | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Batching Tank 80°C, 103 kPa, 316 SS | | | | | 4,790 | | 2,750 | 7,540 |
| | | Primary Reactors 150°C, | | | | | | | | |
| | | 462 kPa, Carpenter 20 | | | | | 49,470 | | 53,790 | 103,260 |
| | | Secondary Reactors 190°C, | | | | | | | | |
| | | 1200 kPa, Carpenter 20 | | | | | 32,980 | | 35,860 | 68,840 |
| | | Flash Drum, 110°C, 316 SS | | | | | 12,490 | | 4,400 | 16,890 |
| | | Flash Drum, 110°C, 316 SS | | | | | 12,490 | | 4,400 | 16,890 |
| | | Neutralization Tank 80°C, 316 SS | | | | | 1,420 | | 500 | 1,920 |
| | | Screw Conveyor | | | | | 6,410 | | 3,800 | 10,210 |
| | | Screw Conveyor | | | | | 4,220 | | 2,500 | 6,720 |
| | | Screw Conveyor | | | | | 4,220 | | 2,500 | 6,720 |
| | | Bucket Elevator | | | | | 20,080 | | 10,000 | 30,080 |
| | | Screw Conveyor | | | | | 4,220 | | 2,500 | 6,720 |
| | | Screw Conveyor | | | | | 2,200 | | 1,300 | 3,500 |
| | | Pumps | | | | | 21,720 | | 9,000 | 30,720 |
| | | Press | | | | | 247,020 | | 137,000 | 384,020 |
| | | | | | | | | | | |
| | | Total: | | | | | 423,730 | | 270,300 | 694,030 |
| | | | | | | | | | | |
| CONTINUED ON SHEET: | | | | | | | | | | |

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems
0700 Hemicellulose and Lignin Extraction
LOCATION
OWNER

EST. BY
COST/C.F.
COST/S.F.
JOB NO.(OWNER)

PROJECT NO.
COST/
DATE
DATE DUE



19
19

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Pulping tank, 80°C, 103 kPa, MS | | | | | 23,420 | | 8,250 | 31,670 |
| | | Surge tank, 80°C, 103 kPa, MS | | | | | 2,870 | | 1,650 | 4,520 |
| | | Lignin precipitation tank, 80°C 103 kPa, 304 SS | | | | | 78,060 | | 27,500 | 105,560 |
| | | Lignin storage tank, 80°C, 103 kPa, MS | | | | | 5,740 | | 3,300 | 9,040 |
| | | Hemicellulose tank 80°C 103 kPa, 304 SS | | | | | 76,580 | | 44,000 | 120,580 |
| | | Lignin storage bin, 50°C 103 kPa, MS | | | | | 57,430 | | 33,000 | 90,430 |
| | | Screw conveyor | | | | | 4,220 | | 2,500 | 6,720 |
| | | Flight conveyor | | | | | 1,900 | | 8,800 | 10,700 |
| | | Screw conveyor | | | | | 8,160 | | 5,000 | 13,160 |
| | | Flight conveyor | | | | | 3,450 | | 1,900 | 5,350 |
| | | Screw conveyor | | | | | 8,440 | | 5,000 | 13,440 |
| | | Screw conveyor | | | | | 1,010 | | 600 | 1,610 |
| | | Pumps | | | | | 43,440 | | 18,000 | 61,440 |
| | | Vari-nip press filter | | | | | 222,170 | | 325,000 | 547,170 |
| | | Rotary drum filter | | | | | 239,200 | | 100,000 | 339,200 |
| | | Cyclone | | | | | 5,460 | | 3,500 | 8,960 |
| | | Spray dryer system | | | | | 270,060 | | 341,000 | 611,060 |
| | | Scrubber | | | | | 19,180 | | 10,000 | 29,180 |
| | | Pump | | | | | 7,240 | | 3,000 | 10,240 |
| CONTINUED ON SHEET | | Total | | | | | 1,078,030 | | 942,000 | 2,020,030 |

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Process Systems — EST. BY _____ — PROJECT NO. _____

0800 Yeast Production — COST/C.F. _____ — COST/ _____ — SHEET 11 OF 14

LOCATION _____ — COST/S.F. _____ — DATE _____ 19____

OWNER _____ — JOB NO.(OWNER) _____ — DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Nutrient tank 30°C, 103 kPa MS | | | | | 1,870 | | 660 | 2,530 |
| | | Yeast production tank | | | | | 166,640 | | 58,700 | 225,340 |
| | | Yeast storage bin 50°C, 103 kPa MS | | | | | 6,880 | | 3,960 | 10,840 |
| | | Screw conveyor | | | | | 8,160 | | 5,000 | 13,160 |
| | | Pumps | | | | | 28,960 | | 12,000 | 40,960 |
| | | Filter | | | | | 11,230 | | 20,000 | 31,230 |
| | | Centrifuge | | | | | 87,050 | | 155,000 | 242,050 |
| | | | | | | | | | | |
| | | Total: | | | | | 310,790 | | 255,320 | 566,110 |
| | | | | | | | | | | |
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WLW-4 77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Utilities EST. BY _____ PROJECT NO. _____

_____ COST/C.F. _____ COST/ _____ SHEET 12 OF 14

LOCATION _____ COST/S.F. _____ DATE _____ 19____

OWNER _____ JOB NO.(OWNER) _____ DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Compressed air system | | | | | | | | 80,000 |
| | | Boiler system | | | | | | | | 700,000 |
| | | Cooling tower system | | | | | | | | 200,000 |
| | | Electrical (off-site, site, miscellaneous, utility related) | | | | | | | | 683,000 |
| | | Misc. (1% of total direct cost) | | | | | | | | 300,000 |
| | | | | | | | | | | 1,963,000 |
| CONTINUED ON SHEET: | | | | | | | | | | |

WLW-4-77

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Controls and Computer EST. BY _____ PROJECT NO. _____

COST/C.F. _____ COST/ _____ SHEET 13 OF 14

LOCATION _____ COST/S.F. _____ DATE _____ 19____

OWNER _____ JOB NO.(OWNER) _____ DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Printer | | | | | | | | 90,000 |
| | | Dynamic graphic console | | | | | | | | 150,000 |
| | | Host computer systems | | | | | | | | 200,000 |
| | | Installations | | | | | | | | 132,000 |
| | | Miscellaneous controls (buildings, | | | | | | | | |
| | | services, utilities, process not | | | | | | | | |
| | | covered by module) | | | | | | | | 438,000 |
| | | | | | | | | | | |
| | | | | | | | | | Total: | 1,010,000 |
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| CONTINUED ON SHEET | | | | | | | | | | |

W. L. WARDROP & ASSOCIATES LTD.
ENGINEERING CONSULTANTS

**ESTIMATING SHEET**

☐ PRELIMINARY
☐ BUDGET
☐ FINAL

PROJECT Wastewater Treatment EST. BY _____ PROJECT NO. _____

_____ COST/C.F. _____ COST/ _____ SHEET 14 OF 14

LOCATION _____ COST/S.F. _____ DATE _____ 19____

OWNER _____ JOB NO.(OWNER) _____ DATE DUE _____ 19____

| CODE NO. | ITEM | DESCRIPTION | UNIT | QTY. | INSTALLATION & INDIRECTS | | | EQUIPMENT | | TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | HRS. | $/HR. | TOTAL | UNIT PRICE | TOTAL | |
| FORWARD FROM SHEET: | | | | | | | | | | |
| | | Aerated cell earthwork | | | | | | | | 53,000 |
| | | Liner | | | | | | | | 45,900 |
| | | Aeration System | | | | | | | | 69,000 |
| | | Settling cell - earthwork | | | | | | | | 14,100 |
| | | Piping and valves | | | | | | | | 12,000 |
| | | Site work | | | | | | | | 6,000 |
| | | | | | | | | | Total: | 200,000 |
| CONTINUED ON SHEET: | | | | | | | | | | |

WLW-4-77

LABORATORY EQUIPMENT COST BREAKDOWN

BIOLOGICAL LAB

| Equipment | Quantity | Price |
|---|---|---|
| pH Meter | 2 | $3,400 |
| Accessories | | 600 |
| Balance | 4 | 14,200 |
| Accessories | | 2,000 |
| Centrifuge | 1 | 10,000 |
| Accessories | | 7,000 |
| Coulter Counter | 1 | 30,000 |
| Microscope | 1 | 25,000 |
| Accessories | | 3,000 |
| Colony Counter | 1 | 21,000 |
| Laminar Flow Chamber | 1 | 10,000 |
| Accessories | | 1,000 |
| Anaerobic Chamber | 1 | 20,000 |
| Autoclave | 1 | 50,000 |
| Freeze Dryer | 1 | 10,000 |
| Accessories | | 1,000 |
| Fermenters | | |
| -(Automatic) | 2 | 60,000 |
| -(Glass) | 10 | 10,000 |
| Votex | 1 | 200 |
| Column Chromatograph | 1 | 10,000 |
| Accessories | | 2,000 |
| Heating Bath Circulator | 2 | 5,000 |
| Shakers | | |
| (temp. control) | 2 | 10,000 |
| (w/out temp. control) | 1 | 5,000 |
| BOD Bottle Probe | 2 | 1,200 |
| $O_2$ Analyzer | 1 | 1,300 |
| Accessories | | 400 |
| Incubator/Refrigerator | 4 | 10,000 |

Laboratory Equipment Cost Breakdown
Biological Lab - continued

| Equipment | Quantity | Price |
|---|---|---|
| Cold Room Install'n | 1 | 22,000 |
| Dish Washer | 1 | 20,000 |
| Glasswares | | 10,000 |
| Hot Plates & Stirrers | | 5,000 |
| Freezer | 1 | 6,000 |
| Office Equipment | | 5,000 |
| Miscellaneous | | 50,000 |

| | |
|---|---|
| Total Equipment Cost | $441,100 |
| Bench & Laboratory Furnishing | $75,000 |
| Indirect Cost | $121,900 |
| Total Cost | $640,000 |

Laboratory Equipment Cost Breakdown
- continued -

CHEMICAL LABORATORY

| Equipment | Quantity | Price |
|---|---|---|
| pH Meter | 2 | $3,500 |
| Accessories | | 800 |
| Balance | 4 | 16,200 |
| Centrifuge | 1 | 500 |
| Accessories | | 200 |
| Filter | 1 | 120 |
| Oven | | |
| (Gravity Convection) | 2 | 2,000 |
| (Vacuum) | 1 | 2,000 |
| HPLC (High Performance Liquid Chromatograph) | 1 | 100,000 |
| UV Spectrophotometer | | |
| Single Beam | 1 | 1,500 |
| Accessories | | 200 |
| Double Beam | 1 | 20,000 |
| Accessories | | 2,000 |
| Glucose Analyzer | 1 | 6,500 |
| Gas Chromatograph | 1 | 25,000 |
| Accessories | | 5,000 |
| Evaporator | | 1,500 |
| I.R. (Double Beam) | | 16,000 |
| Accessories | | 4,000 |
| Electrophoresis | 1 | 10,000 |
| Kjeldahl Unit | 2 | 2,000 |
| Viscometer | 1 | 1,500 |
| Accessories | | 1,000 |
| Glasswares | | 10,000 |
| Refrigerator | 2 | 6,000 |

Laboratory Equipment Cost Breakdown
Chemical Laboratory - continued

| Equipment | Quantity | Price |
|---|---|---|
| Water Bath Circulator | 2 | 5,000 |
| Dishwasher | 1 | 20,000 |
| Hot Plates & Stirrers | | 5,000 |
| Atomic Absorption (Double Beam) | 1 | 40,000 |
| Accessories | | 5,000 |
| Furnace | 1 | 1,500 |
| Distilled Water Equipment, Steam Heated Model | | 8,000 |
| Demineralizer | | 7,000 |
| Office Equipment | | 5,000 |
| Miscellaneous | | 50,000 |

| | |
|---|---|
| Total Equipment Cost | $384,000 |
| Bench & Laboratory Furnishing | $75,000 |
| Indirect Cost | $134,000 |
| Total Cost | $593,000 |

APPENDIX H
MARKETS

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INTRODUCTION

Markets exist for the major products of the pilot plant. Minor products will provide material for new process development with the excess burned in the plant boilers to recover the heat energy.

The products of the pilot plant can be divided into three distinct groups:

- Primary products produced by the plant as designed.
- Secondary products which are being commercially produced using primary products as raw materials.
- Potential products which are presently in the research and pilot testing field but which will fill a large volume or high value market when placed in production using primary products as raw materials.

Plant Primary Products

Primary products include ethanol, lignin, pentose sugars, fusel oil and single cell protein (yeast). The quantity of each of these products for a 10 tonne per day pilot plant are shown below.

| | PILOT PLANT | |
|---|---|---|
| | Acid Hydrolysis | Enzyme Hydrolysis |
| Dry Wood, tonne/day | 10 | 10 |
| Ethanol, L/day | 3,000 | 3,250 |
| Lignin, tonne/day | 2.26 | 2.26 |
| Pentose Sugars, tonne/day | 0.65 | 0.65 |
| Fusel oil, L/day | 13.4 | 14.5 |
| Single cell protein, kg/day | 290 | 330 |

The quantities above are maximum quantities based on the optimum configuration for each product.

## Ethanol

Ethanol will be the largest quantity product produced by the pilot plant. As an industrial solvent, it is used mostly in the printing trade and as a solvent or dilutant in compounded products including cosmetics, perfumes, paints, inks, adhesives and automotive products.

There are five major sources for ethanol in Canada, these are:

1. Commercial Alcohols Ltd.
2. Ontario Paper Company Ltd.
3. Mohawk Oil Company Ltd.
4. Imported Alcohol
5. Technical Alcohol from beverage distilleries.

Commercial Alcohols Ltd. of Montreal is the largest producer of industrial ethanol in Canada. They produce approximately 68.1 million liters of anhydrous ethanol (United States Pharmicopea spec.) from ethylene. Originally, they operated a fermentative alcohol plant using spent sulfite liquor until process changes at the Canadian International Paper Company mill changed the substrate characteristics.

Ontario Paper Company in Thorold, Ontario, produce 5.9 million liters per year of 95% ethanol based on fermentation of spent sulfite liquor. Stillage is evaporated and burned in a recovery boiler.

Technical alcohol produced as a by-product of beverage distilling is relatively small in volume compared to the two major producers.

Mohawk Oil Company has recently converted a beverage distillery to the production of anhydrous ethanol for gasohol from barley. Production will be 5.2 million liters per year with a potential capacity of 6.4 million liters per year.

A new industrial alcohol plant (Tem Bec) in Temiskaming, Quebec is being planned and should be operating in 1983-84 with production of 18 million liters per year and a capacity of 27 million liters per year.

Alcohol imports for 1980 were all from the United States and amounted to 285,000 liters.

The Canadian market for ethanol is 79 million litres/year. This includes all medical and pharmaceutical uses but also includes at least 6.8 million litres of ethanol of lower grade than will be produced by the pilot plant. The maximum annual production from the pilot plant of 1 072 500 L/yr can probably be sold within the existing low grade industrial alcohol market in Canada as it represents less than 16% of this market.

The price for anhydrous denatured ethanol in tank car lots FOB Edmonton is estimated at $0.77 per litre, based on the drum price less a drumming charge.

The annual sales value of ethanol at maximum production rate is $800,000, in bulk, FOB an Edmonton plant site.

Gasohol Market

Ethanol can be mixed with gasoline to produce a fuel which can be used in unmodified gasoline engines. The most common gasoline ethanol blend contains 10% ethanol and 90% gasoline, commonly called gasohol. The octane rating of alcohol is 110 compared with 92 for regular gasoline. This allows gasohol to be a high-test unleaded fuel (octane 94) without the addition of antiknock additives.

Recent tests (Bell Telephone Co. - Illinois, U.S.A.) indicate mileage with gasohol is slightly better than with gasoline. The energy value of alcohol is lower than gasoline by volume, however, the higher octane improves the utilization of the available energy.

In an attempt to promote development of alternative fuels, the Government of Manitoba has removed all provincial taxes on gasohol provided it contains at least 10% alcohol of at least 99% purity, produced in Canada from biomass. The ethanol produced by the pilot plant meets these criteria.

The present Manitoba provincial tax on gasoline is 5.7¢/litre. Since gasohol contains 10% ethanol, one litre of ethanol produces ten litres of gasohol, resulting in a tax saving of $0.57/litre. The ethanol replaces an equal volume of gasoline which is worth, wholesale, $0.28/litre, excluding federal tax.

The value of ethanol for gasohol is composed of tax value, fuel value less transportation to Manitoba.

| | Manitoba | Alberta |
|---|---|---|
| Tax value | $0.57 | $0.0 |
| Fuel value | 0.28 | 0.28 |
| Transportation to Manitoba | -0.078 | 0.0 |
| TOTAL | $0.772 | $0.28 |

The price of ethanol used for gasohol and the value of ethanol for industrial use in Edmonton are equal. It can be assumed that they will remain equal unless the tax advantage in Manitoba is removed or restricted. All factors involved in the production, distribution and tax are directly proportional to the cost of energy. The total production can probably be sold under contract to an existing sales organization. This would reduce the cost of marketing by using an existing sales organization.

## Fusel Oil

Annual production of fusel oil at the pilot plant could reach 3000 to 4000 litres based on 250 production days. This volume does not warrant the cost of marketing or separating the components. A typical analysis of fusel oil from corn fermentation is presented below:

| Component | Fusel Oil Additives: % |
|---|---|
| N Propyl Alcohol | 11 |
| Isobutyl Alcohol | 11 |
| Active Amyl Alcohol | 21 |
| Isoamyl Alcohol | 54 |
| Hexyl Alcohol | 3 |
| | 100 |

Fusel oil from the pilot plant can be mixed with ethanol if the ethanol is being used for gasohol or for some industrial uses. This use would yield the best price obtainable with no additional marketing cost. If it cannot be combined and sold with ethanol, it should be burned in the pilot plant boiler to recover the heat value.

## Lignin

Lignin is the second largest product produced by the pilot plant. The quality of the lignin produced and its selling price, are determined by the pretreatment and hydrolysis processes used. If the lignin is subjected to acid hydrolysis, it is not saleable as a chemical raw material and must be burned in the plant boilers to recover its heat value of 3.3¢/kg.

If lignin is extracted before acid hydrolysis, or after enzyme hydrolysis, the lignin will be the highest quality obtainable. In fact, no market value can be placed on this product because, at present, lignin of this quality is not available in commercial quantities. This material can probably be used as the starting point for all of the lignin based secondary products and tertiary products.

Lignin is produced as a byproduct of paper manufacture. The quantity presently produced would amount to thousands of tonnes per day, most of which is concentrated and burned in recovery boilers. A small portion of the lignin produced is sold or used for secondary products.

| | Production kg | Price $/kg | Sales Volume |
|---|---|---|---|
| Lignin liquor (1979) | 36 209 474 | 0.039 | $1 423 000 |
| Lignin pitch (1979) | 3 678 796 | 0.202 | 745 000 |
| Lignosulphonate (1979) | 1 337 849 | 0.258 | 345 000 |
| | 41 226 119 | | $2 513 000 |

Lignin produced by the pilot plant will probably sell well above the price of existing markets for use in pharmaceutical, vanillin and glue manufacture. The price should not drop below that for lignin pitch because, in spite of its high quality, it can be used as a binder material for animal feed pellets and for foundry molding sand, both uses require substantial volumes on a long term basis.

The three major suppliers of lignin products in North America are American Can Co. in Green Bay, Wisconsin, Ligisol Ltd., a subsidiary of Reed Paper Company of Montreal, and Georgia Pacific Corp. Together, these three companies have two-thirds of the North American lignin market. The total volume of lignin sold amounts to 450 000 tonnes/year. Most lignin is modified to produce marketable products. Modified lignins are used as dispersing agents in concrete, particle board manufacture, boiler water treatment chemicals, and as drilling mud viscosity improvers. It is also the raw material for vanillin production. Lignin is used as a binder for animal feeds and an additive for phenolic plastics, rubber and asphalt.

American Can Co. produces fifteen separate lignin-based products, produced largely from their Green Bay mill's spent sulfite liquor. They purchase 4.5 tonnes of soft wood spent sulfite liquor (50% solids) per month for use in one particular product. They pay 5.46¢ U.S/kg for this product.

Once the quality of lignin produced by the pilot plant has been established, the total lignin production of the plant can best be sold under contract to one of the major lignin marketers, users or pharmaceutical producers.

Pentose Sugars

Pentose sugars are not fermentable to ethanol by the normal yeast strains used in ethanol production. Some microorganisms have been identified which can produce ethanol from pentose and the pilot plant will utilize these. The remaining pentose sugars can be used for single-cell protein production or concentrated to molasses for use as cattle feed. The lowest value use would be concentration and burning in the plant boiler to recover the heat value. Recovery of pentose sugars not only produces a marketable product but reduces the load on the waste treatment system.

The present price of molasses in bulk FOB Minneapolis-St. Paul is $127/tonne. The price in bulk FOB Winnipeg is $143/tonne.

## Single Cell Protein

A market presently exists for single cell protein as a human food additive. A much larger market exists, at a lower price, for single cell protein as a protein source for animal feeds. The pilot plant can produce food quality product from poplar if lignin has been extracted.

Yeast (Candida utilis) can be grown under aerobic conditions using pentose sugars with ammonia as a nitrogen source. The product can find a ready market as a food additive if the colour and flavour are acceptable. Three plants are presently producing food quality yeast in the United States. Two yeast plants are operating on spent sulphite liquor, one at Boise Cascade in Salem, Oregon, the other at Monarch Paper in Reinlander, Wisconsin. The third major producer of food quality yeast is Pure Cultures Inc. of Hutching, Minnesota, a subsidiary of Amoco, which produces a high quality yeast using alcohol as the substrate.

The Boise Cascade plant has two fermenters processing about 25 L/s of spent sulphite liquor from Hemlock to produce 2.4 million kilograms per year of dried yeast. Retention time in the fermenters is about three hours and the process removes 50% of the sugars present. Yeast from the fermenter is centrifuged, washed, centrifuged and spray dried. The waste liquor is evaporated and burned in a recovery boiler.

The Reinlander Paper plant has a Waldorf Process fermenter which uses 9 L/s of spent sulphite liquor, from Tamrack and Spruce, containing 8-10% sugars to produce 3.6 million kilograms of yeast per year. Retention time in the fermenters is roughly three hours and the removal of sugars is almost complete. Yeast from the fermenters is centrifuged, washed, centrifuged and spray dried.

| Processor | Production kg/year | Price $/kg (assumed) | Sales Volume $/year |
|---|---|---|---|
| Boise Cascade Inc. | 2 700 000 | 1.10 | 3 000 000 |
| Monarch Paper Inc. | 3 600 000 | (1.10) | 4 000 000 |
| Pure Cultures Inc. | 2 700 000 | 1.76 | 4 800 000 |

If yeast is not salable as food grade, it can be sold for use in animal feeds. A large market exists for yeast as animal feed largely based on nitrogen content. The major sources of this material are breweries and distilleries. Dried brewers yeast is presently (September 7/81) selling for $502/tonne in 18 000 kg carload lots.

## Secondary Products

Secondary products include vanillin, lignosulphonates, furfural, butanol, acetone and xylitol.

### Vanillin

An oversupply of vanillin exists presently in North America. However, the quality of lignin produced by the pilot plant may be valuable as an auxiliary feedstock for existing plants. The price of vanillin in 50 kg drums is $14.40/kg. A raw material which could improve the yield in existing plants would draw a good price and would probably be considered for an exclusive contract.

Vanillin is manufactured from lignin by oxidation in a caustic soda solution after which it is extracted and purified to provide commercial vanillin. Approximately 80% of the North American market is supplied by Ontario Paper Company, Thorold, Ontario. Productive capacity at present in North America is 5200 tonnes per year while sales are 4000 tonnes per year. The oversupply has been caused by a slowing of demand in the pharmaceutical use which was predicted to maintain 10% annual growth but has recently maintained 3% annual growth. The pharmaceutical industry consumes 40% of the vanillin supply for the manufacture of drugs. It is expected that no new productive capacity will be brought on line for approximately five years. Ontario Paper Company are interested in testing the lignin produced at the demonstration plant.

North American Vanillin Market:

| | |
|---|---|
| Ontario Paper Company | 1600 tonnes + 1600 tonnes to Europe |
| Other Suppliers (Bouregard, Rayonier) | 2400 tonnes |
| Total | 4000 tonnes |

Furfural

The market for furfural in Canada is too small to justify a production facility. However, pentose sugar molasses could be saleable as a concentrated feedstock for existing furfural plants to increase capacity or yield.

Furfural is produced from nonfood agricultural residues, i.e. oat hulls, rice hulls, corn cobs and wood wastes. Pentosans are hydrolyzed to pentoses which are cyclodehydrated to furfural. In all cases, raw material is treated with strong inorganic acid and the furfural is steam distilled leaving ash, lignin and the remaining cellulose. The distillate is refined further to produce commercial furfural.

Commercial uses of furfural in order of their importance are:

1. Selective solvent in the petroleum, resin and chemical industries.

2. Extractive distillation agent in petrochemical production.

3. Resin intermediate and reactive solvent.

As a selective solvent, it is used for extraction of aromatic and unsaturated compounds. Materials treated include lubricating oils, diesel fuel, catalytic cycle stock and virgin feed stock for crackers.

In extractive distillation, it alters the relative volatilities of $C_4$ hydrocarbons making possible the economic separation of butadiene.

As a resin intermediate and reactive solvent, it is used in phenolic thermosetting resins to reduce viscosity of the resin for molding but reacting to form part of the set resin.

No furfural is produced in Canada at present, but total imports in 1979 amounted to 304 tonnes with a commercial value of $399 000 or $1.31/kg.

Major manufacturers in the United States are Quaker Oats Co. and Ashland Chemical Co.

Butanol

The present market for butanol in Canada is too small to justify a production facility, however, pentose sugar molasses can be fermented to butanol and acetone.

Butanol was manufactured by fermentation of molasses or grain with acetone as a byproduct. Since 1951, synthetic production has increased to become the predominant method of manufacture. The oxo process is the most common process used today. This process involves treatment of propylene with carbon monoxide and with hydrogen to produce butylaldelydes which are reduced in the form of butyl alcohol. Butanol is used largely in the coatings industry as various esters. Butanol with butyl acetate has been used in nitrocellulose lacquers while butyl acrylate is used in acrylic latex paints. Small quantites are used as plasticizers and in hydraulic fluids as a coupling agent.

There is no Canadian production of butanol at present. All imports in 1980 were from the United States and amount to 209 tonnes/year of butanol with a value of $127 000 or $0.608/kg.

Xylitol

No market presently exists in North America for Xylitol.

Xylitol is a saccharide alcohol manufactured from xylose and used as a dietetic sweetener. The product was first developed in Europe and appeared to have tremendous market potential in North America. Test results published in 1980 in England showed that it caused tumours in laboratory animals. This development completely stopped process development at Pfeizer Inc. and halted design on an 11 000 tonne per year plant for Hoffman-La Roche Inc.

## Products with High Potential Markets

Research directed into the following areas has the potential of developing large volume markets for the products, specifically lignin, of a commercial scale poplar chemical plant and should strengthen the wood-based chemical industry in Alberta. Potential lignin products are more fully detailed in the attachment.

Lignin-based adhesives for plywood and particle board manufacture would provide a high volume moderately priced product based on a steady long-term market. Most work up to now has been based on lignin from paper manufacture. Availability of unreacted lignin will allow researchers to tailor the modifications to provide the desired properties.

Lignin-based polymers and plastics will provide a large market if they can capture even a minor portion of the industrial plastics market. Most research in this area has been on lignin-based urethanes. Availability of unreacted lignin may allow the completion of this work.

Ethylene can be manufactured from ethanol. A new catalyst allows a six-fold improvement in efficiency and a 97% selectivity for ethylene. At present, this process is not economic, however, changes in petroleum refining could change the availability of this basic polymer feedstock.

Fine chemicals and pharmaceuticals, while not requiring large volumes of primary products, produce specialized, high value products. The presence of a commercial scale plant could create the nucleus of a high technology, wood-based chemical industry in Alberta.

Conclusions

1. A sound market exists for ethanol either as industrial alcohol in Alberta or as gasohol in Manitoba. The market is large enough to accept all of the pilot plant production. Marketing is best done by long term contract.

2. The high quality lignin which will be produced at the pilot plant is not presently available in commercial quantiies. This will provide an excellent base material for production of vanillin, pharmaceuticals and other high value products. Marketing is best done by contract with major lignin marketers or lignin users. Research into the use of this form of lingin holds potential for an Alberta wood based chemical industry.

3. Single cell protein should be suitable for human consumption. Alternately, it will find a ready local market in animal feed formulations.

4. The remaining products should be used for product research but are in quantities too small to warrant the cost of marketing. The pilot plant boilers should be equipped to burn fusel oil and possibly concentrated lignin solutions.

# LIGNIN MARKET

## Introduction

Wood and agricultural crop residues have a large potential as a source of chemicals. The uses of cellulosics as a source of chemicals has been reviewed recently [48]. The chemical composition of these raw materials is as follows:

| | % Cellulose | % Hemicellulose | % Lignin |
|---|---|---|---|
| Poplar | 56 | 22 | 18 |
| Conifers | 47 | 22 | 29 |
| Wheat Straw | 39 | 38 | 18 |
| Corn Cobs | 41 | 41 | 17 |

From this table we see that these feedstocks range from about 40% to over 50% cellulose, which may be used for a variety of uses: paper, chemical derivatives or hydrolysis to sugars. Such sugars form the basis of a fermentation industry to make alcohols and a variety of other products. Hemicellulose, which is principally pentosans, is useful either for fermentation to alcohol or for furfural, xylitol and other chemicals. Hemicellulose content of agricultural crop residues is much higher than in the woods, about 40% compared with about 22%. Lignin which constitutes about 18% of poplar or of agricultural crop residues, enjoys to date a limited market. Of $530 million in wood-based chemicals sold in the United Stated in 1977, $90 million was either lignin or lignin derivatives, about 17% of the total.[49] Nearly all of this was a by-product of the sulphite pulping industry. About one-third of this lignin was converted to vanillin, the remainder, mainly as lignosulphonates, was used used as an adhesive binder, as a particle board resin extender, in oil well drilling mud viscosity control and as a surface active agent.

The quantities of lignin available from a poplar alcohol facility are quite considerable. The Alberta Poplar Chemical Plant should make 3000 litres of ethanol/day and there should be available a maximum of 1.8 tonnes of lignin or over 600 tons of lignin per year. A full scale facility making 100,000 litres/day alcohol -- the "standard" Brazilian size -- would generate 20,000 tons lignin per annum. This quantity of lignin cannot be ignored, it may be a nuisance, a fuel or a source of profit. This statement reviews current market-oriented work in progress.

## Characteristics of Lignin from the Alberta Poplar Chemical Plant

There are three possible sources of lignin from the processes installed at the Alberta Poplar Chemical Plant:

i) Purified lignin from extraction of uncatalysed autohydrolysis pretreated fibre.

ii) Lignin from enzyme hydrolysis, before or after fermentation.

iii) Acid hydrolysis lignin.

Acid hydrolysis lignin is a highly condensed char which, at the moment, could be considered only for fuel. It is difficult to visualize what other uses could be developed for it.

Purified lignin obtained by alkaline extraction of uncatalysed autohydrolysis pretreated fibre is a light tar free flowing powder of well defined chemical composition.[50] It is the most valuable form of lignin for medical uses and for a variety of derivatives.

Lignin obtained from enzyme hydrolysis also has the characteristics of the purified lignin just mentioned but is contaminated with fibre and with protein from enzymes and fungi. It may be purified for use by solution in alkali and reprecipitation, whereupon it would be expected to be just as useful as the pretreatment lignin.

It should be stressed that the properties of purified lignin and enzyme hydrolysis lignin depend upon the conditions of autohydrolysis pretreatment.[51] The presence of acid catalysis in autohydrolysis causes condensation to a black, intractable tar.

## Lignin as Fuel

When clean and dry, acid hydrolysis lignin has a fairly high heat value of 25,800 KJ/kg.[52] In relation to the total alcohol production process, we may use the estimate [5] of 35 million KJ/day for processing 10 tonnes of aspen/day in the pilot plant. The lignin content of the aspen has 46 million KJ, so the alcohol process would be energy self-sufficient with the recovery of 75% of the lignin in dry form. This is not truly a net value, since acid hydrolysis lignin requires washing or neutralizing to get rid of acid, and then moisture removal to some low value, say 10%, to enable its use as fuel.

This may or may not be an economic procedure on this small scale, but undoubtedly would pay on the large scale. The lignin would be worth $60. to $100. per tonne based on its heat content alone, and in a 100,000 litre/day plant would be worth $1.2 to $2.0 million per year. This calculation sets a minimum value for the lignin, regardless of what other uses may develop, the fuel value is not inconsiderable.

## Vanillin and Related Compounds

The largest single lignin market to date is in making vanillin and related compounds. Vanillin is made by the oxidation of the lignin in sulphite spent lignin, a diminishing resource. Autohydrolysis lignin can be used just as satisfactorily in making vanillin.

Poplar lignin contains two different structures so that oxidation produces syringealdehyde along with the vanillin. These need to be separated. There is a good market for both aldehydes.

Vanillin is best known as a flavouring agent for ice cream and other food products. It is the basis, however, for a whole range of pharmaceuticals, from ethyl vanillate, which is a potent fungicide used for athlete's foot, to L-dopa (L-dioayphenylalanine) used in the treatment of Parkinson's disease. Pharmaceutical uses of vanillin derivatives are probably a greater source of income than flavouring uses. Syringealdehyde is also acceptable and apparently preferred for a number of pharmaceutical uses.

While we understand that the market is quite good just now, and shows some growth, one cannot expect unlimited growth of vanillin and its derivatives. The single Canadian producer, The Ontario Paper Company in Thorold, Ontario, is probably the world's largest producer, making about 6,000,000 lbs./year (2,700 tonnes/year). It is believed that any expansion of production can only come from autohydrolysis lignin, but one full scale alcohol plant with 10,000 to 20,000 tonnes/year of lignin by-product could saturate this market for some years. However, it is a high value product and should not be ignored.

Medical Uses of Lignin - Lignin as Dietary Fibre

Recent research in Toronto has shown that Aspen autohydrolysis lignin has properties of dietary fibre and modifies bile acid and blood composition in a highly favourable manner. In animals, aspen autohydrolysis lignin prevented gallstone formation on high cholesterol lithogenic diets.[53] This work, the prevention of gallstones, is now being extended to a selected population of humans at risk.

Dietary fibre is also known to have benefits in the preventing of cancer of the colon. Animal experiments using poplar autohydrolysis lignin for its effects on cancer of the colon are proceeding in a number of research centres, and it is hoped to begin human trials in the not-too-distant future.

Poplar autohydrolysis lignin has also been shown, in animals, to reduce blood cholesterol significantly.[54] This may have application in heart disease prevention and treatment.

Poplar autohydrolysis lignin is also known to be an anti-oxident and it is now being tested to neutralize the effects of dietary nitrosamines, suspected of contributing to stomach cancer.

The availability of poplar autohydrolysis lignin, following the pioneering work of J.H. Lora [55] and the collaboration of the University of Toronto and Stake Technology in preparing several hundred kilograms, has opened up a whole new area of medical research. This is based on the hypothesis of Dr. Ruth Kay, of the Toronto Western Hospital, that lignin is the active component of dietary fibres.

It is obvious that if it can be established that poplar autohydrolysis lignin is valuable in the prevention and treatment of such a variety of widespread ailments as cancer of the colon, stomach cancer, heart disease and gallstones, the market will be very substantial. Besides, it will have merit that goes far beyond market considerations.

## Lignin as Adhesive and Binder

A reasonably substantial use of lignin is as a binder for foundry cores and as an adhesive in the manufacture of linoleum, ceramics and other products. A related use is pelleting chicken feed.

Recent trials have suggested that poplar autohydrolysis lignin may be used as an effective binder or adhesive in making fibreboard. This work is still proceeding and is hopeful.

Research carried out at Forintek, initially with kraft lignin and subsequently with poplar autohydrolysis lignin, has shown that lignin modified with formaldehyde is useful as an extender for particleboard glues. It is hoped, from this work, to achieve two additional goals: to be able to use properly modified lignin as a substitute for present particleboard glues; and to formulate liquid glues for plywood manufacture. The successful accomplishment of these objectives would open a significant market for poplar autohydrolysis lignin. It would be expected that the availability of such lignin in large quantity at an acceptable price, would do much to advance this particular use.

## Lignin in the Oil Industry

Lignosulphonates have long been used in the oil industry and are well accepted for drilling mud viscosity control. As the sulphite pulp industry phases out, the oil industry will look for alternative sources of lignosulphonates. Poplar autohydrolysis lignin can be readily sulphonated and can be expected to claim a portion of this market.

Partly in consequence of their familiarity with the product, lignosulphonates are being used by most of the oil companies in their studies of enhanced oil recovery. As this market develops, it can be expected that sulphonated poplar autohydrolysis lignin can take a share of it.

## Lignin Polyurethanes and Other Polymers

Goldstein has estimated [56] that 95% of U.S. polymer output could be derived from wood and that about one-third of this could come from the lignin. He suggested lignin as a source of polystyrene, polyesters and nylon. He speculated lignin-based production of over 6 million tons of plastics, derived from 15 million tons of wood, (the Canadian pulp and paper industry uses about 32 million tonnes of wood per year). To date no lignin has found its way into polyurethane, polystyrene, polyester or other such markets. Most research reported to date has been on polyurethanes,[57] of which about one million tonnes was used in the United States in 1979. It is apparent that such markets are large enough to be worth a special effort to capture at least a part of them as a means of marketing lignin.

A difficulty with lignin for use as a base for urethanes is a low content of hydroxyl groups. This can be increased by one of two methods, condensation with formaldehyde to form hydroxymethylene derivatives, and demethylation. The successful achievement of high hydroxyl content lignin would open the door to these markets. It should be noted that urethanes are now used as adhesives and core-builders, two uses where lignin has traditionally found a place. The conversion of lignin to polyurethanes would consolidate and extend these traditional markets.

The largest use for polyurethanes is as foam, representing over 80% of polyurethane consumption. The remainder is used in rubber-like compounds and surface coating, as well as adhesives and sealants. The largest adhesive use was in foundries, 75 million pounds per year, with a projected growth rate of 8 to 10% per annum.[58] This suggests a market well worth pursuing.

## Phenols from Lignin

There is a great shortage of phenols now, particularly for phenol-formaldehyde resins and the dibasic phenol resorcinol, which is important in rubber manufacture as well as in high-strength waterproof plywood glue, is now at a very high price. The structure of lignin, including the aromatic ring bearing one or two methoxyl groups, suggests that it should be a ready source of phenols. This has been the subject of much research over many years but the demethylation of lignin has proven to be far more difficult than appears on paper. There is no commercial process for phenols from lignin, however, this is an active area of research [59] and the promise is there. Someday, someone will find the key to the demethylation of lignin and open another substantial market. As with other uses, the ready availability of poplar autohydrolysis lignin at an acceptable price will do much to stimulate such research and progress it to success.

# APPENDIX I
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